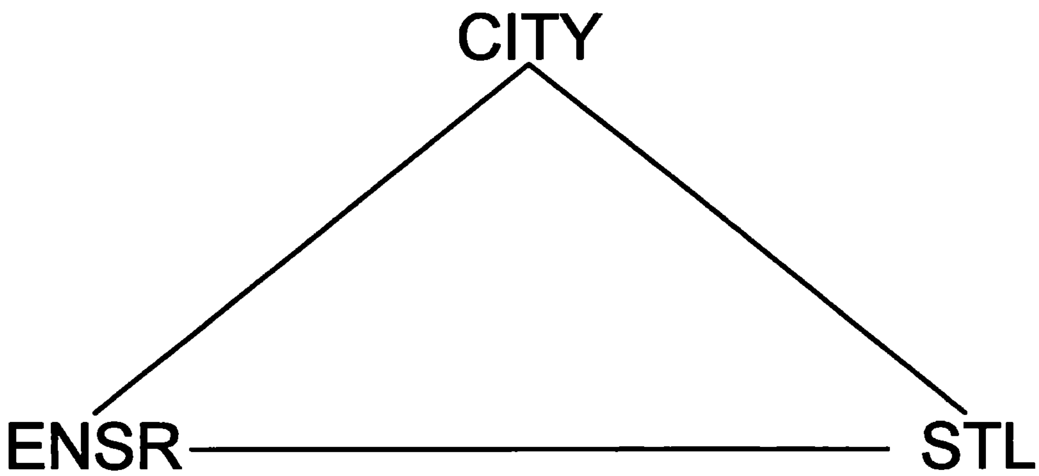
2002 SAMPLING PLAN

REILLY TAR & CHEMICAL CORP.
N.P.L. SITE
ST. LOUIS PARK, MINNESOTA

SUBMITTED OCTOBER 31, 2001



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October 2001

REILLY TAR AND CHEMICAL CORPORATION

N.P.L. SITE

ST. LOUIS PARK, MINNESOTA

SITE MANAGEMENT PLAN

INTRODUCTION

Groundwater in the City of St. Louis Park, Minnesota, has been found to contain polynuclear aromatic hydrocarbons (PAH) as a result of activities at a coal-tar distillation and wood preserving plant (Site) operated from 1917 to 1972. Numerous previous studies have identified PAHs in various aquifers beneath St. Louis Park and adjacent communities.

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Agency (MPCA), the Minnesota Department of Health (MDH), the City of St. Louis Park (City), and Reilly Industries, Inc. (formerly Reilly Tar & Chemical Corporation - Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into a Consent Decree, include the following concentration levels:

	Advisory Level	**Drinking Water Criteria**
Sum of benzo(a)pyrene and dibenz(a,h) anthracene	3.0 ng/l*	5.6 ng/l
Carcinogenic PAH	15 ng/l	28 ng/l
Other PAH	175 ng/l	280 ng/l

*** or the lowest concentration that can be quantified, whichever is greater**

In conjunction with the implementation of remedial measures to limit the spread of PAH, granular activated carbon (GAC) treatment systems have been installed to treat water from City wells (identified - SLP) 4, 10 and 15. Further provisions of a Remedial Action Plan (RAP) call for long-term monitoring of the influent and effluent of the GAC treatment systems and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH in the groundwater. The analytical data will be used to evaluate water quality by comparing the levels of PAH found in the various samples with historical water quality data and with water quality criteria established in the Consent Decree-RAP. The specific objectives of the monitoring program, and therefore, the intended end use of the data vary slightly for the different aquifers being monitored in accordance with the Consent Decree-RAP.

The objective of the GAC treatment system monitoring is to assess and evaluate the performance of the treatment systems. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the Consent Decree-RAP. Based on these comparisons, decisions will be made on: 1) system operations (e.g., when the carbon should be replaced), and 2) cessation of the treatment systems, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of monitoring the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells and any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH, is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the Site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the Consent Decree-RAP.

If any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23, then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the Consent Decree-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking wells, private or industrial wells, and monitoring wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control systems, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the Consent Decree-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23). Water level data will be used to evaluate groundwater flow patterns in the Prairie du Chien-Jordan Aquifer.

The objectives of monitoring St. Peter Aquifer wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating a gradient control system installed at W410 in 1990, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data, to drinking water cessation criteria for well W410, and to drinking water criteria established in the Consent Decree-RAP. Water level data will be used to evaluate groundwater flow patterns in the St. Peter Aquifer.

The objective of monitoring the Drift-Platteville Aquifer wells is to monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control systems. Groundwater analytical data will be used to compare levels of PAH in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the Consent Decree-RAP for PAH. Water level data will be used to evaluate groundwater flow patterns in the Drift-Platteville Aquifer.

The Site Management Plan (Plan) outlines the scope of work to be performed in order to monitor the groundwater in the St. Louis Park, Minnesota, area in accordance with the Consent Decree-RAP related to the Reilly N.P.L. Site and in accordance with the Agencies' October 3, 2000, and October 19, 2000, letters. The Agencies' letters identified the wells to be sampled in the Drift, Platteville, St. Peter and Prairie du Chien-Jordan Aquifers. Wells in the Mount Simon, Hinckley and Ironton-Galesville Aquifers will be sampled in accordance with CD-RAP criteria. Included in this Plan are: 1) the identity of wells to be monitored, 2) the schedule for groundwater monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting. A GAC treatment system has been constructed to treat water from well W23 and the Drift-Platteville Aquifer source control wells prior to discharge to surface water. However, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the Consent Decree-RAP. Similarly, a GAC treatment system has been constructed to treat water from well SLP4 prior to discharge to the municipal water supply system; however, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the Consent Decree-RAP.

The time period covered by this Plan is from January 1, 2002, or the date of its acceptance and approval by the Agencies whichever is later, to December 31, 2002. The next subsequent Sampling Plan (RAP Section 3.3) will be submitted by October 31, 2002 covering the 2003 calendar year.

This Plan incorporates the requirements of RAP Sections 3.2, 3.3, 4.3, 5.1, 7.3, 8.1.3, 9.1.3, 9.2.3, 9.3.3, and 9.6 and the Agencies' October 3 and 19, 2000, letters. Some of the monitoring required under these RAP Sections has already taken place in accordance with previous Sampling Plans.

MONITORING SCHEDULE

The monitoring schedule outlined in this Plan indicates the starting criteria and the frequencies of monitoring as outlined in the RAP and the Agencies' October 3 and 19, 2000, letters to determine when the GAC treatment system and wells are monitored (Tables 1 and 2). In general, the monitoring schedule will allow economies of scale in the field and in the laboratory by grouping the various monitoring events described by the RAP and the Agencies' October 3, 2000, and October 19, 2000, letters as much as possible. Samples will be collected within the frequency indicated on Tables 1 and 2.

Tables 1 and 2 summarize the GAC system/groundwater monitoring schedule for the period through December 31, 2002, and represent the minimum monitoring program that is likely to occur during the year. However, additional monitoring will take place if the drinking water criteria established in the Consent Decree-RAP is exceeded in samples collected from:

- the GAC treatment system treated water
- active municipal drinking water wells
- the Prairie du Chien Aquifer sentry wells (wells SLP6, W48, W119 or W413)

This additional monitoring is described in Sections 4 and 12 of the RAP, and are reproduced in Appendix A of this Plan.

Figures A-2 through A-6 identify the location wells to be monitored in each of the five aquifers. The duration of field sampling events will depend on the number and type of wells to be monitored. For estimating purposes, Drift and Platteville Aquifer monitoring wells typically are monitored at a rate of five to 10 wells per day, St. Peter Aquifer monitoring wells typically are monitored at a rate of five wells per day, and Prairie du Chien Aquifer monitoring wells typically require two to four hours or more per well to monitor.

TABLE 1

Sampling Plan GAC Treatment System Monitoring Schedule[a]

RAP Section	Sampling Points	Start of Monitoring	Sampling Frequency	Analyses[b]
4.3.1(C)	Treated water (TRTD)	Date of plan approval	Quarterly	PAH (ppt)[c]
4.3.3(D)	Feed water (FEED)	Date of plan approval	Annually	PAH (ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH (ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625

a This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 2002. Sections 4 and 12 of the CD-RAP outline the additional monitoring that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring.

b Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one every 10 samples or fewer. Treated water will be duplicated at a rate of 100 percent. Feed water duplicate samples will be collected and analyzed at a frequency of one per 10 samples.

c ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

TABLE 2

Sampling Plan Groundwater Monitoring Schedule[a]

Source of Water	CD-RAP References	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]
Mt. Simon-Hinckley Aquifer	5.1	SLP11, SLP12, SLP13, SLP17	Date of plan approval	Annually	PAH (ppt)[d]
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH (ppt)
Ironton-Galesville Aquifer	6.1.4	W105	Date of plan approval	Every even numbered year[h]	PAH (ppt)
	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH (ppt)
Prairie du Chien-Jordan Aquifer	Table 1[e]	SLP6, W48, W119, W413	Date of plan approval	Quarterly	PAH (ppt)
	Table 1	SLP4, SLP10 or SLP15, W23, W29, W40, W70, W401, W402, W403, E2, E3, E7, E13, E15	Date of plan approval	Annually	PAH (ppt)
	Table 1	E4, SLP5, SLP8, W32	Date of plan approval	Semi-annually	Water level monitoring[f]
	Table 1	H6, MTKA6, SLP7 or SLP9, SLP14, SLP16, W405 or W406[g]	Date of plan approval	Every even numbered year	PAH (ppt)
St. Peter Aquifer	Table 2[e]	SLP3, W24, W33, W122, W133, W410, W411, W412	Date of plan approval	Semi-annually	PAH (ppt)
	Table 2	W409	Date of plan approval	Semi-annually	8270C PAH

TABLE 2

Sampling Plan Groundwater Monitoring Schedule[a]

Source of Water	CD-RAP References	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]
	Table 2	P116, W129, W408	Date of plan approval	Semi-annually	Water level monitoring
Platteville Aquifer	9.2.3	W421	Date of plan approval	Quarterly	8270C PAH[J]
	Table 2	W20, W27, W101, W131, W143, W426, W428, W431, W433, W434, W437, W438	Date of plan approval	Semi-annually	8270C PAH
	Table 2	W1, W18, W19, W100, W120, W121, W124, W130, W424	Date of plan approval	Semi-annually	Water level monitoring
Drift Aquifer	9.1.3	W420	Date of plan approval	Quarterly	8270C PAH
	Table 2	P109, P112, P307, P308, P309, P310, P311, P312, W11, W117, W136, W422, W427, W439	Date of plan approval	Semi-annually	8270C PAH
	Table 2	P47, W2, W10, W15, W116, W128, W135	Date of plan approval	Semi-annually	Water level monitoring

A This schedule does not include certain contingencies (e g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 2002 Section 12 of the CD-RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring Field blanks will be collected at a frequency of one for every 10 samples or fewer, and one duplicate sample will be collected for every 10 samples.

B Sampling points are located on the maps shown in Figures A-2 through A-6. Letter prefixes to well codes are defined as follows:

- W 4-inch Monitoring Well
- P Monitoring Piezometer
- SLP St. Louis Park supply well
- E Edina supply well

TABLE 2

Sampling Plan Groundwater Monitoring Schedule[a]

Source of Water	CD-RAP References	Sampling Points[b]	Start of Monitoring	Sampling Frequency	Analyses

H Hopkins supply well

MTK Minnetonka supply well

C Lists of parameters and descriptions of the methods for analysis of PAH and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analyses, except for those wells which prove to be inaccessible for such measurements

D ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

e Wells will be sampled in accordance with Table 2 from the Agencies' October 3, 2000, letter, or Table 1 from the Agencies' October 19, 2000, letter, in lieu of the requirements given in the Consent Decree. Wells W19 and W130 were listed in the Agencies' Table 2 for both the Drift and Platteville Aquifers. Wells W19 and W130 are Platteville Aquifer wells.

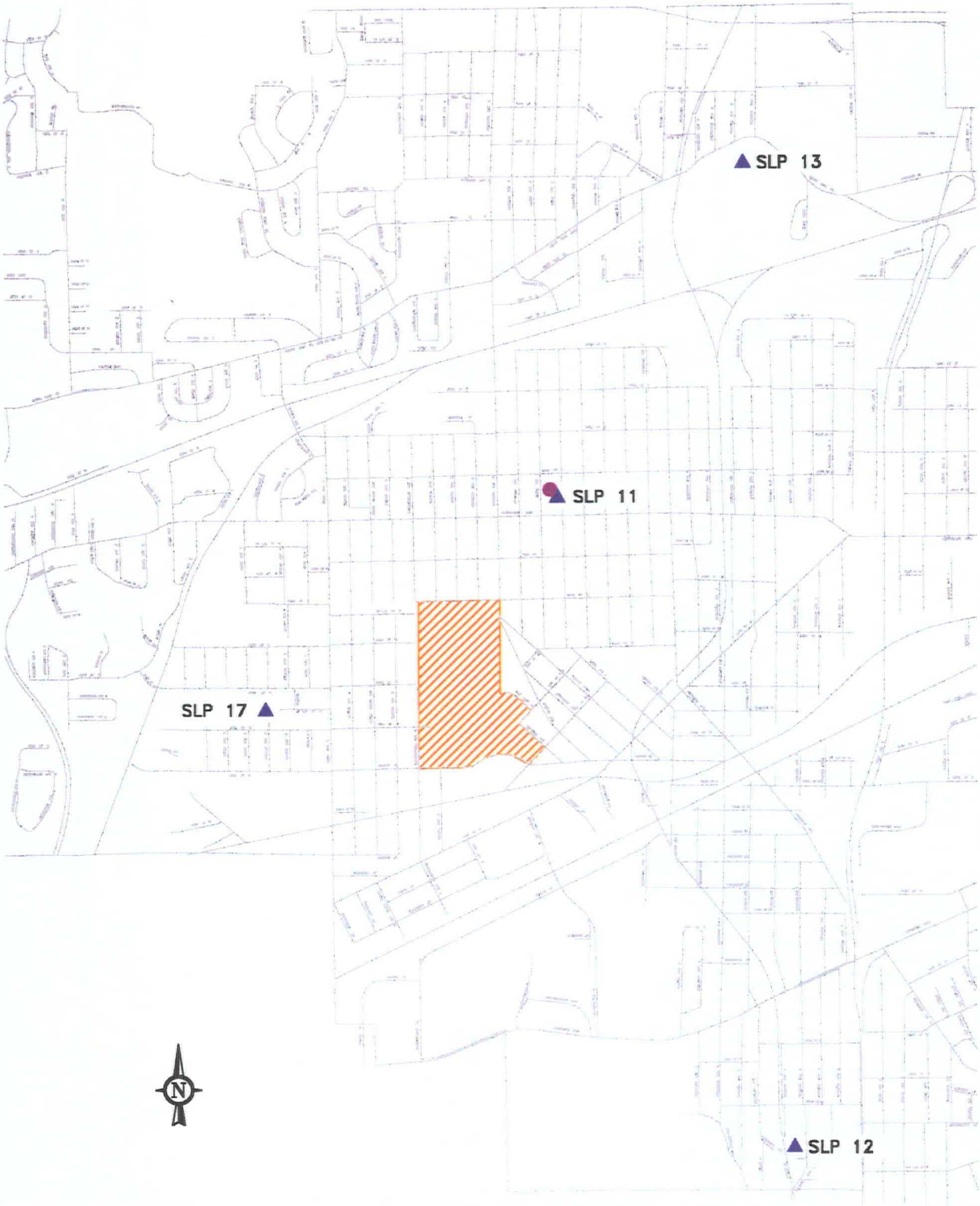
F Water levels will be measured semi-annually at these wells in addition to the sampled wells, except for those wells that prove to be inaccessible for such measurements.

G W405 = American Hardware Mutual, W406 = Minikahda Golf Course.

H Wells sampled every two years shall alternate the season the well is sampled. For example, SLP14 will be sampled in fall 2002, spring 2004, fall 2006, etc.

I ppb = parts per billion. This signifies analysis by full scan 8270C.

J 8270C signifies USEPA SW846 Method 8270C for 16 PAH compounds.



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REILLY SITE

LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

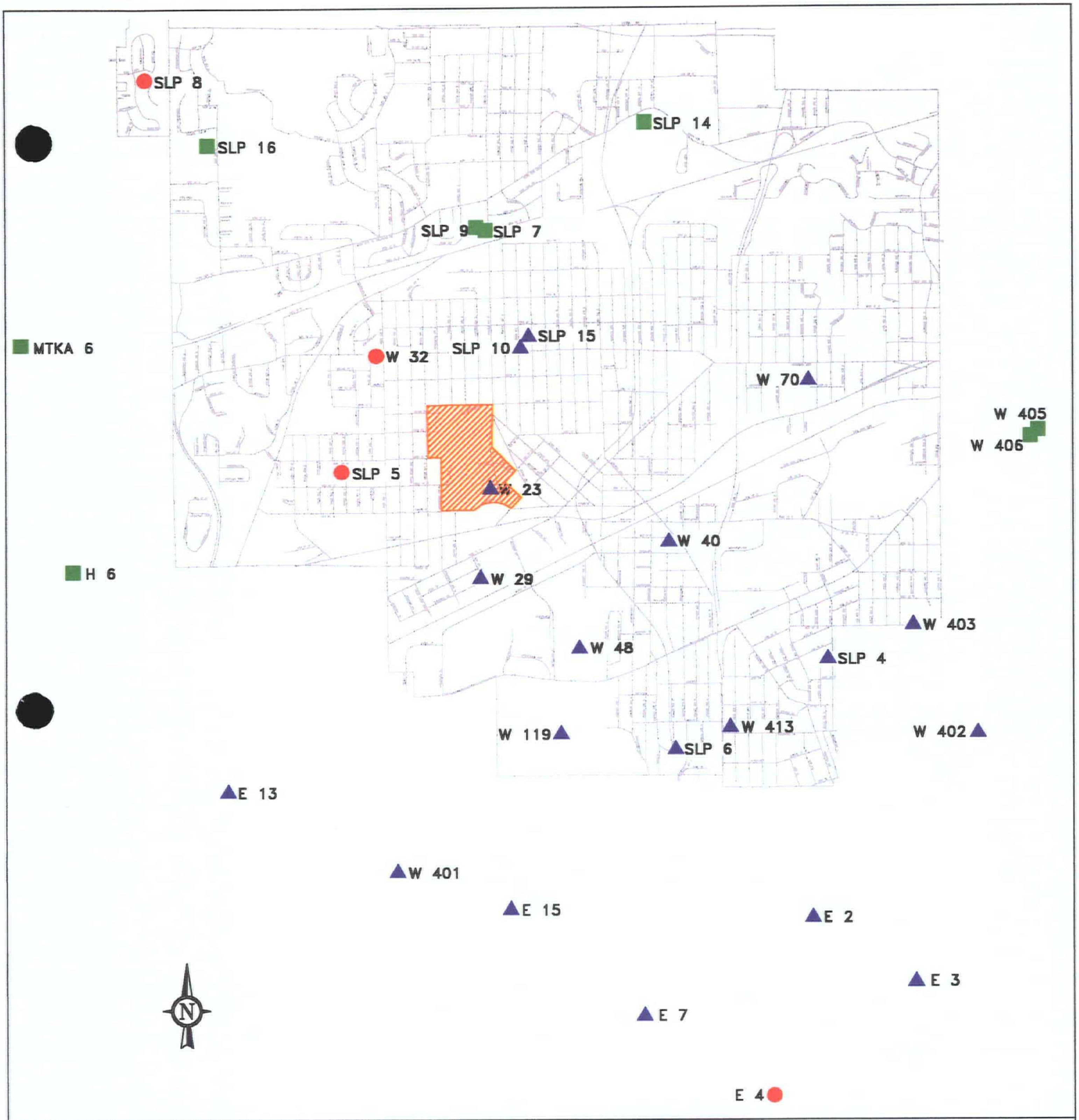
LOCATION OF GAC TREATMENT PLANT

FIGURE A-2

LOCATION OF MOUNT SIMON HINKLEY AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN: C. BOEHM	DATE: 10/30/01	PROJECT NO.: 01620-013	REV:
FILE No.: FIG A-2 MSH.DWG	CHECKED: WMG		



REILLY SITE

LOCATION OF MONITORING WELL THAT IS SAMPLED ANNUALLY, SEMIANUALLY, OR QUARTERLY

LOCATION OF MONITORING WELL THAT IS SAMPLED IN EVEN NUMBER YEARS ONLY (I.E. WELL WILL BE SAMPLED IN 2002, 2004, ETC.)

LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

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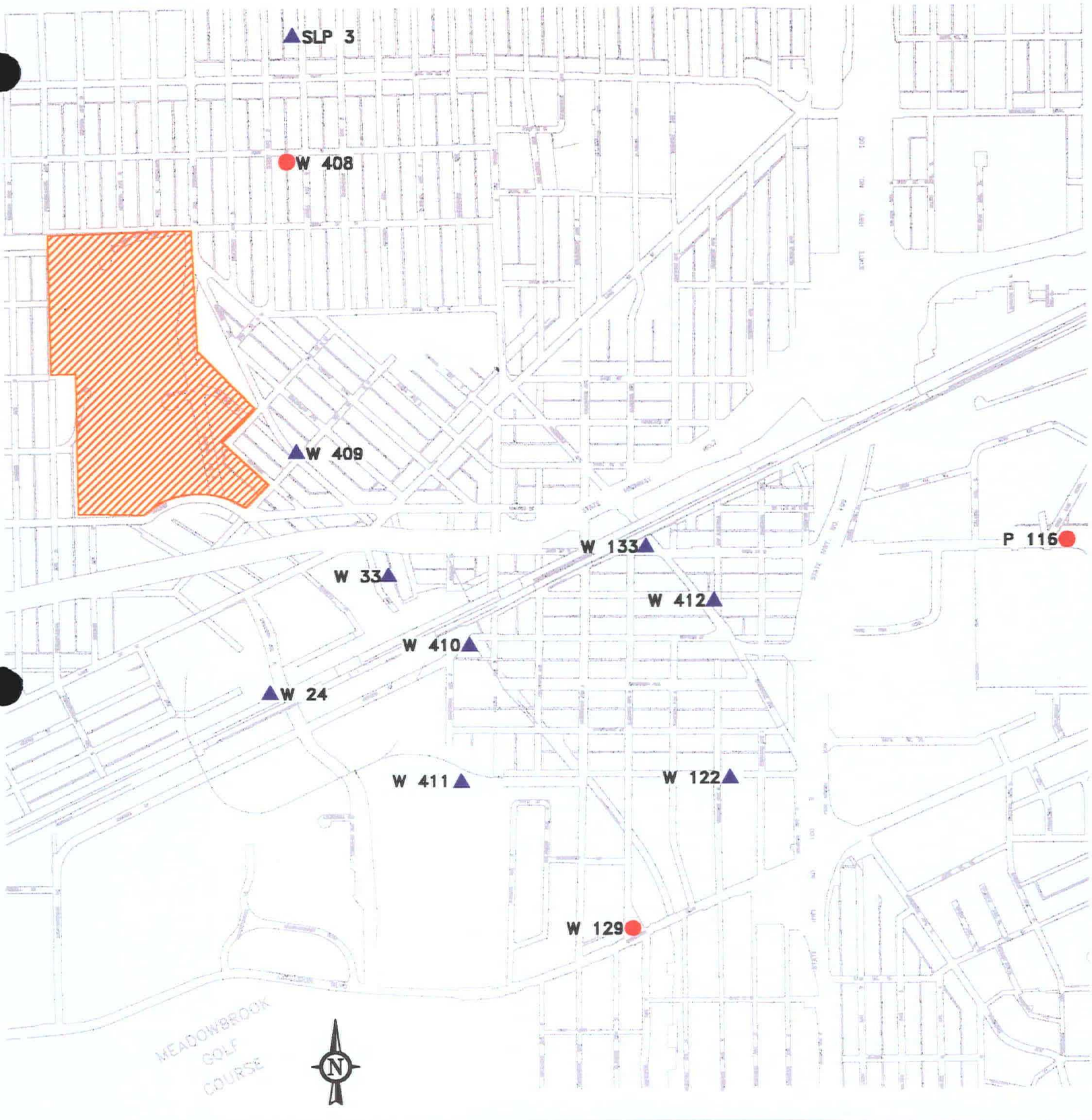
FIGURE A–3

LOCATION OF PRAIRIE DU CHEIN–JORDAN AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN: C. BOEHM	DATE: 10/30/01	PROJECT No.: 01620–013	REV:
FILE No.: FIG A–3 PCJ.DWG	CHECKED: WMG		



REILLY SITE

▲ LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

● LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

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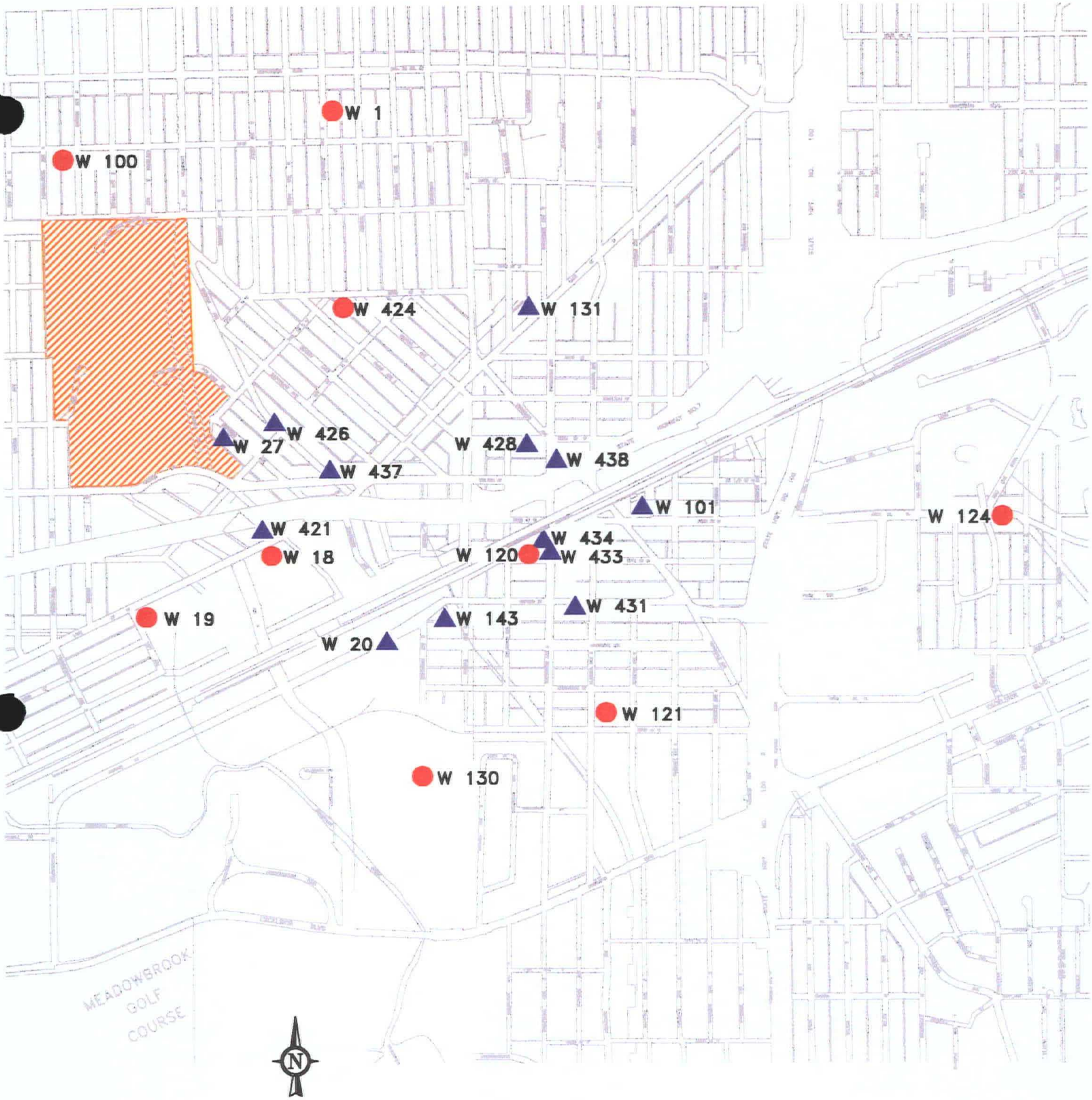
FIGURE A-4

LOCATION OF ST. PETER AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN: C. BOEHM	DATE: 10/30/01	PROJECT No.: 01620-013	REV:
FILE No.: FIG A-4 STP.DWG	CHECKED: WMG		



REILLY SITE

LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

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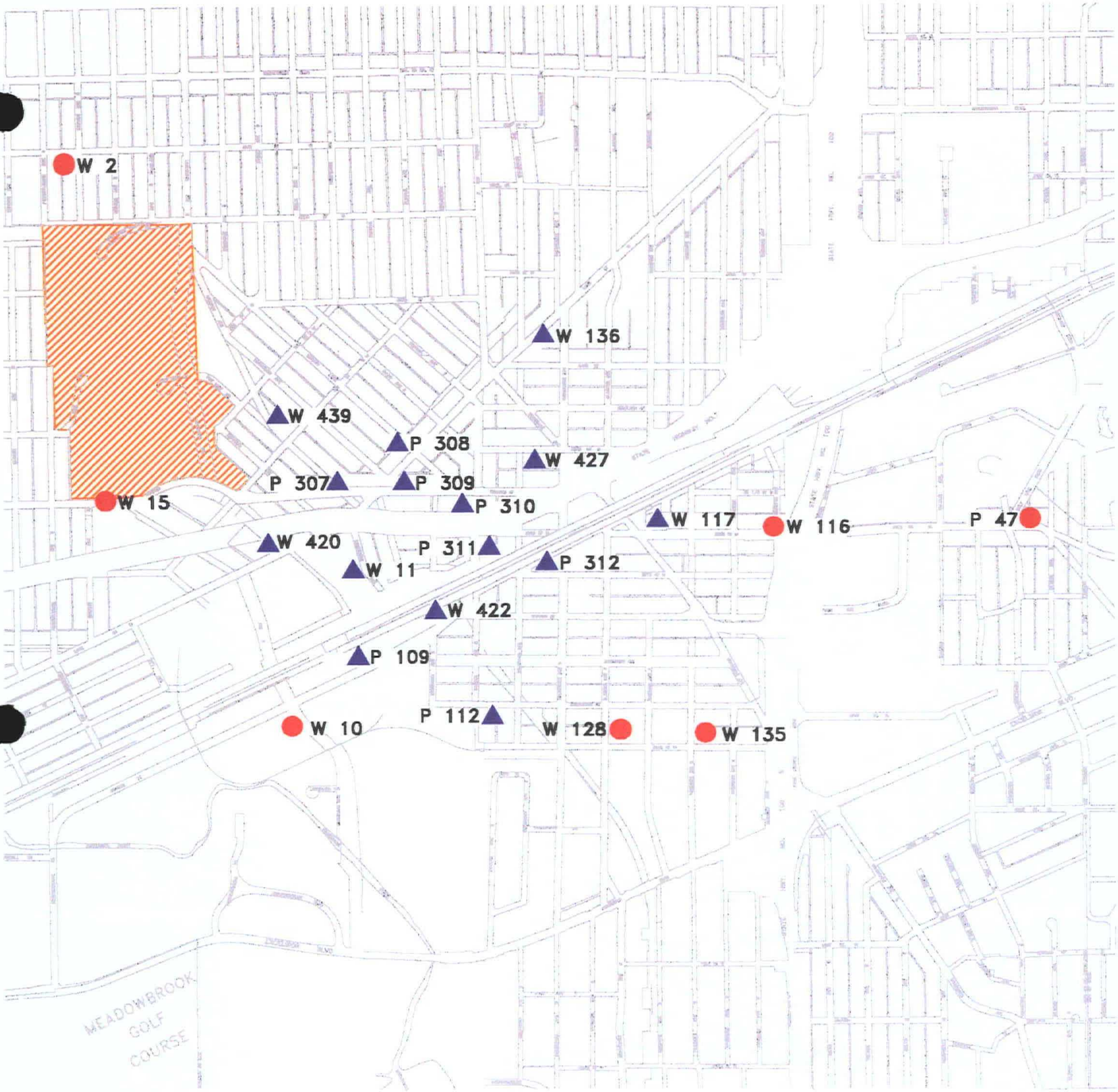
FIGURE A-5

LOCATION OF PLATTEVILLE AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN:	C. BOEHM	DATE:	10/30/01	PROJECT No.:	REV:
FILE No.:	FIG A-5 PLV.DWG	CHECKED:	WMG	01620-013	



REILLY SITE

LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

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FIGURE A-6

LOCATION OF DRIFT AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN:	C. BOEHM	DATE:	10/30/01	PROJECT No.: 01620-013	REV:
FILE No.:	FIG A-6 DRI.DWG	CHECKED:	WMG		

GROUNDWATER SAMPLING PROCEDURES

An important distinction is made between the sampling procedures for active pumping wells (e.g. municipal wells) and for non-pumping monitoring wells. Active pumping wells are used on a regular basis, have dedicated pumps and associated plumbing, and have sample taps for collecting samples. Non-pumping monitoring wells may be new, or may have not been pumped for several years, and most require pumping and associated equipment for sampling. Another distinction is that the active pumping wells are typically located inside buildings whereas non-pumping monitoring wells are not.

With these considerations in mind, this Plan has been developed so that the groundwater monitoring program in each aquifer meets the requirements and intent of the RAP. Groundwater monitoring will be conducted in accordance with the procedures given in the Quality Assurance Project Plan (QAPP), and with Minnesota Pollution Control Agency guidelines entitled "Development of Sampling Plans, Protocols and Reports", January 1995.

Water Level Measurements

Water level measurements will be made using electric tapes or weighted steel tapes. Water level measurements using steel tapes will be made by suspending a known length of tape in the well so that the bottom end of the tape is below the water level. The lower portion of tape will be coated with blue chalk that exhibits a noticeable color change when wetted. The water level measurement will be obtained by subtracting the length of wetted tape from the total length of tape suspended below the measuring point of each well.

Using the electric tape, the probe at the end of the tape will be lowered slowly in the well until contact with the water is made. Because of surface tension, readings of the water level made when the probe enters the water will differ from readings made when the probe leaves the water, thus breaking surface tension. To standardize these measurements, the second reading will always be used (i.e. the reading made when the probe leaves the water).

Water level measurement made for the purpose of defining groundwater flow patterns in a particular aquifer may be performed independently from groundwater sampling, as a discrete event so as not to last more than two days. The wells will be revisited for sampling, and measurements to determine the volume of water in the well will be made at that time.

Sample Collection at Active Pumping Wells

At active pumping wells, the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. An electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered. The only exception to this is the GAC treatment system monitoring under RAP Section 4.3 that includes treated water monitoring.

If it cannot be determined that a well has been pumping at some time during the 24-hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

Sample Collection at Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in the purging and sampling of the monitoring wells and piezometers, flexibility in the approach to sample retrieval is necessary. This Plan proposes that the sampling team be given latitude in the selection of purge/sample equipment and procedures necessary to compete the monitoring task.

Table 2 specifies the monitoring of monitor wells W24 and W70 that are equipped with an operable dedicated submersible pump. Well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with parameter monitoring established herein.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a non-dedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH, and specific conductance of the purge water will be monitored using a Horiba water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or Teflon bailer and a new length of nylon or polyester rope.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without pre-rinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the sampling port or bailer and filled completely, and the cap securely tightened. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

The discharge from purging monitoring wells will be handled in accordance with the

Contingency Plan (Appendix B). In general, if a visible sheen can be seen on the water surface, the discharge will be routed to the sanitary sewer. Otherwise, the storm sewer or surface discharge will be used. Non-dedicated groundwater sampling or monitoring equipment that comes in contact with the groundwater will be decontaminated between uses, as described in the QAPP.

ANALYTICAL PROGRAM

Tables 1 and 2 show the groundwater monitoring summary for the coming year. Indicated on the tables are the analyses required. Details of all analytical methodology can be found in the QAPP and its appendices. All analyses will be performed at SevernTrent Laboratories (STL), formerly Quanterra) Incorporated, Arvada, Colorado, laboratory. STL has agreed to provide a turnaround time of 30-working days from the receipt of samples to the submittal of analytical reports. The laboratory will notify the City if it cannot meet this turnaround time.

Groundwater monitoring will include two methods of PAH analyses depending upon the anticipated PAH concentration levels. Low-Level (nanograms per liter or part per trillion) PAH analyses will be performed utilizing selected ion monitoring (SIM) gas chromatography mass spectrometry (GC/MS). This method will be used to analyze samples from drinking water wells and from other wells for which the RAP requires drinking water criteria to be enforced (e.g. St. Peter Aquifer monitoring wells). This method is designed to analyze samples containing up to 600 nanograms per liter of an individual PAH. With dilution of the sample extract, the effective range of the method can be extended into the microgram per liter range. Specific details of this methodology can be found in Appendix B of the QAPP.

Full scan method SW 846 8270C will be used to measure PAH in the micrograms per liter or part per billion range. PAH analyses, using the full scan method, will be performed on samples from wells that have historically contained elevated PAH concentrations (e.g. greater than 50 micrograms per liter or 50,000 PPT), and on wells that are not subject to the RAP's requirements for meeting drinking water criteria (e.g., Drift-Platteville aquifer wells).

REPORTING

The analytical reporting requirements of the Consent Decree and RAP are identified in Part K of the Consent Decree, and Sections 3.4, 4.3.5, 12.1.1, and 12.1.2 of the RAP. Park K requires Reilly to submit an annual monitoring report on March 15 of each year for data collected from the previous year. This report will contain analytical data and an assessment of data quality as specified in the QAPP. All water level measurements and chemical analyses that have not been presented in previous reports, and interpretive maps and tables, as specified in RAP Section 3.4(B) and (C) will be included in the annual monitoring report. Also, the effectiveness of the source and gradient control well systems in the Drift-Platteville and St. Peter Aquifers will be discussed in the annual report.

The reporting requirements for each aquifer, and for the GAC treatment system, are described below.

GAC Treatment System

RAP Section 4.3.5 requires the City to submit an annual report that presents the results of all monitoring of the GAC treatment system. Analytical results for wellhead water, feed water, and treated water will be included in this report. The report will also describe briefly the operating performance of the GAC treatment system during the previous calendar year. The GAC treatment system annual reports are due each March 15.

Mt. Simon-Hinckley Aquifer

The monitoring data for the Mt. Simon-Hinckley Aquifer will be included in the annual report. In addition to the results of all water level measurements and chemical analyses, the report will contain a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labeled by the location of each well in accordance with RAP Section 3.4(C). Since the Mt. Simon-Hinckley Aquifer wells are monitored on an annual basis, there will be only one sampling event to report.

Ironton-Galesville Aquifer

The monitoring data for well W105, and for any new Ironton-Galesville Aquifer drinking water well installed within one mile of well W23, will be included in the Annual Report

Prairie du Chien-Jordan Aquifer

The monitoring data for the Prairie du Chien-Jordan Aquifer will be included in the annual report. The results of all water level measurements and chemical analyses will be included. For each of the water level measuring periods, a water level contour map will be prepared with elevations labeled at each well. For each sampling event, a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labeled by the location of each well will be prepared in accordance with RAP Section 3.4(C), and a map of the area indicating the extent of PAH above drinking water criteria shall be provided.

St. Peter Aquifer

The monitoring data for the St. Peter Aquifer will be included in the annual report. The results of chemical analyses will be reported and a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labeled by the location of each well will be prepared in accordance with RAP Section 3.4(C). Likewise, the results of water level measurements will be provided and a water level contour map will be prepared with elevations labeled at each well in accordance with RAP Section 3.4(B). In addition, a map of the area indicating the extent of PAH above drinking water criteria shall be provided.

Platteville Aquifer

The monitoring data for the Platteville Aquifer including the results of all water level measurements and chemical analyses, will be presented in the Annual Monitoring Report. A map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labeled by the location of each well, will be prepared in accordance with RAP Section 3.4. The Platteville Aquifer monitoring data will be included in the annual report to support a discussion of the results with respect to the effectiveness of the source and gradient control well systems.

Drift Aquifer

The monitoring data for the Drift Aquifer including the results of all water level measurements and chemical analyses, will be presented in the Annual Monitoring Report. A map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labeled by the location of each well, will be prepared in accordance with RAP Section 3.4. The Drift Aquifer monitoring data will be included in the annual report to support a discussion of the results with respect to the effectiveness of the source and gradient control well systems.

A

APPENDIX A
ADDITIONAL MONITORING REQUIREMENTS

Level or Drinking Water Criterion is exceeded during the first year of operation of the system, Reilly shall immediately notify the Regional Administrator, the Director, and the Commissioner, and shall undertake such additional Monitoring as is required by Section 4.3.2.

(D) Routine Monitoring after two carbon changes shall be quarterly, unless the Regional Administrator, the Director, and the Commissioner determine that the observed service life of the carbon is too short to permit this frequency, in which case the Regional Administrator, the Director and the Commissioner shall notify Reilly of the required Monitoring frequency in accordance with Part G or H of the Consent Decree.

4.3.2. Carbon Replacement Monitoring

(A) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Drinking Water Criterion for Other PAH or exceed the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall collect two additional treated water samples at least 2 Days apart within one week of receiving the results of the exceedance sample. If the analytical results from either one or both of the two additional samples also exceed the Drinking Water Criterion for Other PAH or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(B) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Advisory Level for Other PAH, then Monitoring of treated water shall be conducted immediately according to Section 12.1. If the results of any two samples required by Section 12.1. exceed the Drinking Water Criterion for Other PAH, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(C) If any analytical result from the additional samples taken as required by (A) or (B) above exceeds the Drinking Water Criterion for Other PAH, or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene during either

(1) within one year after the carbon treatment system is placed into service or

(2) within one year after the first carbon change if carbon was changed in the first year of operation of the carbon treatment system,

then Reilly shall conduct the Monitoring program specified in Section 4.6. Reilly shall report the results of the Section 4.6. Monitoring program to the Regional Administrator, the Director and the Commissioner within 7 Days of receiving the analytical data. If the treated water from the carbon treatment system is determined pursuant to Section 4.6. to exceed the Drinking Water Criterion for Other PAH or the Advisory Levels for Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall replace the carbon within 14 Days of making this determination. If the treated water is determined pursuant to Section 4.6. to meet the Drinking Water Criterion for Other PAH and the Advisory Levels for Carcinogenic PAH and the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then normal GAC system operation and Monitoring in accordance with Sections 4.3.1.(B) and

(C) After the first month of operation, Monitoring of feed water shall be performed quarterly until the carbon has been changed twice. If the Regional Administrator, the Director and the Commissioner determine pursuant to Section 4.3.1.(B) that the GAC system is not operating properly, Reilly may, upon receipt of such determination, be required to resume biweekly Monitoring of feed water.

(D) After two carbon changes in the GAC system, feed water shall be Monitored annually.

4.3.4. Extended Monitoring

Treated water from the GAC system shall be sampled and analyzed annually for the extended list of PAH in Part A.2. of Appendix A, using gas chromatography/mass spectroscopy (GC/MS), or other methods approved by the Regional Administrator and the Director. During this extended analysis, any compounds listed in Part A.2. of Appendix A, or any other compounds which are detected with significant peak heights that are not routinely Monitored, shall be identified and, if possible, quantified, using a mass spectral library which contains extensive spectra of PAH compounds, such as the National Bureau of Standards mass spectral library. Reilly shall analyze a sample of treated or feed water once a year for the acid fraction compounds determined by EPA Test Method 625 or by other methods approved by the Regional Administrator and the Director.

CONTINGENT ACTIONS FOR MUNICIPAL DRINKING WATER SUPPLY WELLS

12.1. Contingent Monitoring

12.1.1. Exceedance of Advisory Levels

If the analytical result of any sample taken from an active municipal drinking water well under the Monitoring requirements of Sections 3., 4.3., 5.1., 6.2.1., 7.3., or 8.4. above exceeds an Advisory Level, Reilly shall take another sample within seven Days of receiving the analytical results and analyze this sample. If the results of the second sample are below all of the Advisory Levels, a third sample shall be taken by Reilly within seven Days of receiving the results of the second sample. If the third sample is below all of the Advisory Levels, Monitoring of the affected well shall revert to its normal schedule. If the analytical result of the second or third sample exceeds an Advisory Level but is less than all Drinking Water Criteria, the Regional Administrator, the Director, and the Commissioner shall be notified by Reilly immediately and subsequent samples shall be taken by Reilly monthly until such time as either:

(A) three consecutive samples yield results less than all of the Advisory Levels, in which case the sampling interval shall revert to the level specified for the affected well in Sections 3., 4.3., 5.1., 6.2.1., 7.3., or 8.4. above; or

(B) a sample yields results greater than a Drinking Water Criterion, in which case the requirements of Section 12.1.2., below, apply.

12.1.2. Exceedance of Drinking Water Criteria

(A) If the analytical result of any sample taken from an active municipal drinking water well pursuant to Section 12.1.1 exceeds the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, the Regional Administrator, the Director and the Commissioner shall be immediately notified by Reilly, and another sample shall be taken by Reilly within three Days of receiving the results of the first sample and analyzed. If the analytical result of the second sample is less than all of the Drinking Water Criteria but greater than any Advisory Level, a third sample shall be taken by Reilly within seven Days of receiving the results of the second sample and analyzed. If the results of this third sample are less than all of the Drinking Water Criteria, but greater than any Advisory Level, Reilly shall comply with the monthly sampling frequency specified in Section 12.1.1. above.

(B) If the analytical result of the second or third sample taken pursuant to Section 12.1.2.(A) above is greater than the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, Reilly shall Monitor the well weekly until such time as either: (1) three consecutive samples yield results below all of the Drinking Water Criteria, in which case Monitoring of the well shall revert to the normal schedule (including Advisory Level Monitoring as specified by Section 12.1.1. above, if applicable); or, (2) three consecutive samples yield results above any Drinking Water Criterion, in which case Reilly shall immediately notify the Regional Administrator, the Director and the Commissioner. The Commissioner may then require the affected well to be taken out of service, in which case Reilly shall undertake the contingent actions specified in Section 12.2. below.

12.1.3. Analytical Turn-around Time

All Monitoring conducted pursuant to Section 12.1. shall be on a 21-Day turn-around time basis in accordance with Section 2.8.

B

APPENDIX B
CONTINGENCY

Contingent Actions for Contaminated Water

It is possible that groundwater contaminated with coal tar materials will be encountered during the sample retrieval operations. Groundwater generated during sample retrieval operations will be classified as contaminated if the water exhibits a discernible oil sheen or oil phase. Contaminated water will be pumped to the sanitary sewer if it contains less than 10 percent organic material. Estimates of flow rate, disposal volume and water quality will be established and the Metropolitan Council Environmental Services (MCES) will be informed before the discharge to the sanitary sewer if the estimated flow exceeds 150 gallons per workday from any individual site. Contaminated liquids containing more than 10 percent organic material or failing to receive MCES approval for discharge will be disposed of in accordance with all applicable local, state and federal rules and regulations and Part T of the Consent Decree. Uncontaminated water will be disposed of in the storm sewer or by other means acceptable to the City of St. Louis Park.

The City will be responsible for keeping the Environmental Protection Agency, Minnesota Pollution Control Agency and Reilly Tar & Chemical Corporation informed of all significant actions involving the generation of contaminated groundwater. All actions, decisions and communications by the City, Environmental Protection Agency, Minnesota Pollution Control Agency, and Reilly in dealing with contaminated soils will be in accordance with and subject to the provisions of Parts I, J, and O of the Consent Decree in the Reilly settlement.

2

QUALITY ASSURANCE PROJECT PLAN FOR SAMPLING AND ANALYSIS - GROUNDWATER AND GAC TREATMENT SYSTEM MONITORING

for the

Reilly Industries, Inc.
N.P.L. Site
St. Louis Park, Minnesota

Prepared by:

The City of St. Louis Park
St. Louis Park, Minnesota 55416

Approved by:______________________________ Date: ________________
Larry Penfold, Quality Assurance Manager
STL Denver

Approved by:______________________________ Date: ________________
William Gregg, Project Manager
City of St. Louis Park, MN

Approved by:______________________________ Date: ________________
Quality Assurance Officer
U.S. EPA Region V

Approved by:______________________________ Date: ________________
Remedial Project Manager
U.S. EPA Region V

Approved by:______________________________ Date: ________________
Project Manager
Minnesota Pollution Control Agency

A.1 Table of Contents and Document Control Format

A.1.A Table of Contents

LIST OF APPENDICES

Appendix A: ENSR SOP 7130 "Ground-Water Sample Collection from Monitoring Well"; ENSR SOP 7510 " Packaging and Shipment of Samples", ENSR SOP 7121 "Field and Laboratory Measurement of pH"; ENSR SOP 7123 "Field and Laboratory Measurement of Temperature"; ENSR SOP 7124 "Field and Laboratory Measurement of Specific Conductance"

Appendix B: DEN-QA-0025 "Building Security"; CORP-QA-0010 "Nonconformance and Corrective Action System"; DEN-MS-005 "PAHs by Selective Ion Monitoring for City of St. Louis Park"; DEN-MS-0001DEN "GC/MS Analysis Based on Method 8270C and 625"; DEN-QA-003 "Sample Receipt and Chain of Custody"

LIST OF TABLES

LIST OF FIGURES

A.1.B Document Control Format

Document Control for the Quality Assurance Project Plan (QAPP) serves a two-fold purpose. It is a formal system of activities that ensures that:

1. All participants in the project are promptly informed of revisions of the QAPP

2. All documents generated during the course of the program are accounted for during, and at the end of the project

This QAPP and all SOP documents have the following information on each page:

- Project Name
- QAPP
- Revision Number
- Revision Date
- Section/Element
- Page Number

When any of these documents are revised, the affected pages are reissued to all personnel listed as document holders with updated revision numbers and dates. Issuance of revisions is accompanied by explicit instructions as to which documents or portions of documents have become obsolete.

A.2 Distribution List

Darryl Owens
USEPA
77 West Jackson
Mail Code HSR-6J
Chicago, Illinois 60604

Nile Fellows
Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, Minnesota 55155

Tamra Kress
Reilly Industries
300 North Meridian Street, Suite 1500
Indianapolis, Indiana 46204-1763

Brian Stringer
STL Denver
4955 Yarrow Street
Arvada, Colorado 80002

William Gregg
ENSR
4500 Park Glen Road, Suite 210
St. Louis Park, Minnesota 55416

Michael Rardin
City of St. Louis Park
5005 Minnetonka Boulevard
St. Louis Park, Minnesota 55416-2290

A.3 Project/Task Organization

A.3.A Management Responsibilities

This project is being conducted in accordance with the CD-RAP for the Reilly Tar & Chemical Corporation N.P.L Site in St. Louis Park, Minnesota. The parties to the Consent Decree include the Reilly Industries, Inc. (formerly Reilly Tar & Chemical Corporation - Reilly), City of St. Louis Park, Minnesota (City), United States Environmental Protection Agency (EPA), Minnesota Pollution Control Agency (MPCA), and Minnesota Department of Health (MDH). The responsibility for the overall QA/QC on the project is ENSR *International* (ENSR). Both the City and ENSR are responsible for the completion of the monitoring tasks described in the Sampling Plan and this QAPP. The City is assisted in the retrieval and laboratory analysis of water samples by ENSR and Severn Trent Laboratories, Inc. (STL), respectively. ENSR is responsible for the field sampling QA/QC and will perform the biannual audit of STL.

The EPA and MPCA are responsible for review and approval of the Sampling Plan, including the QAPP. In addition, laboratory and field audits may be completed by appropriate agency representatives. Responsibilities of the key positions in the EPA and MPCA are described below:

- EPA Project Manager (Mr. Darryl Owens): The EPA Project Manager, EPA Region 5, is responsible for the review and approval of the QAPP on behalf of the EPA. Mr. Owens is responsible for obtaining input, as needed from the EPA Quality Assurance Officer, and the EPA Central Regional Laboratory, EPA Region 5.

- MPCA Project Manager (Mr. Nile Fellows): The MPCA Project Manager shall be responsible for review and approval of the Sampling Plan, and review of laboratory and field procedures described in the QAPP.

A.3.B QA Responsibilities

As discussed above, EPA and MPCA Project Managers (Messrs. Owens and Fellows) are responsible for QAPP review and approval. ENSR's Project Manager, Mr. William Gregg, is responsible for data validation and data assessment. The City and the Agencies are responsible for internal and external performance and system audits

A.3.C Field Responsibilities

ENSR and the City will be responsible for the coordination of all field sample collection under direction of the Field Coordinator (ENSR Project Manager). The Sampling Team shall consist of employees of the City and ENSR. The team shall be responsible for sample collection, conducting field measurements (i.e. water level), and maintaining proper decontamination procedures stated in the QAPP. The person responsible for identifying and documenting nonconformance and subsequent corrective action is the ENSR Project Manager.

A.3.D Lab Responsibilities

STL, with analytical facilities in Arvada, Colorado, will be responsible for the coordination and completion of all laboratory groundwater and treatment water analyses for polynuclear aromatic hydrocarbons (PAH) and semi-volatile organic compounds. Responsibilities of the key positions in the organization of STL are described below:

- Laboratory Project Manager: The Laboratory Project Manager is ultimately responsible for all laboratory analyses and is the primary point of contact for issues surrounding this QAPP, resolving technical problems, modifications to SOPs, etc. The Laboratory Project Manager is responsible for the coordination of routine day-to-day project activities including project initiative, status tracking, data review and requests, inquiries and general communication related to the project. Final approval of data packages is the responsibility of the Laboratory Project Manager.

- Operations Manager: The Operations Manager is responsible for oversight of all aspects of log in, preparation, and analysis of samples to ensure that project objectives, requirements and QA/QC criteria are met. The evaluation of data, as it is compiled and organized in accordance with the requirements of the QAAP, is the responsibility of the section supervisors, reporting to the Operations Manager. Additional review, evaluation, and assessment of the data as it pertains to the QAPP is performed by an experienced second party reviewer, also reporting to the Operations Manager.

- Analyst: The Analyst is responsible for the analysis of water samples for the requested parameters utilizing the methods prescribed by the QAPP.

- Technician: The Technician is responsible for sample extraction. This requires practical experience and knowledge in the techniques of liquid - liquid solvent extraction, Kuderna - Danish evaporation, and the quantitative preparation of sample extracts for analysis.

- Quality Assurance Director: The Quality Assurance Manager is responsible for overall quality control oversight, including internal audits and corrective actions. The Quality Assurance Manager supervises an independent QA/QC department and reports directly to the Laboratory Director and Corporate QA Director.

- Sample Control Group Leader: The Sample Control Group Leader is responsible for overseeing the sample control staff in sample receipt by the laboratory. This includes inspection of sample containers and chain of custody records, noting of errors, omissions, or out of control items, and ultimately logging samples into the laboratory database. The Sample Control Group Leader communicates with the Laboratory Project Manager and reports to the Operations Manager.

A.3.E Special Training Requirements/Certification

All ENSR and City personnel working on the project will be properly trained and qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- Organization and lines of communication and authority
- Overview of the Site Management Plan and QAPP
- Documentation requirements
- Decontamination requirements
- Health and Safety considerations

Training of field personnel will be provided by the Field Coordinator or a qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency in the analytical methods to be employed.

A.3.F Project Organization Chart

The project organization shown in Figure A-1 indicates the involvement of the parties to the Consent Decree, as appropriate.

A.4 Problem Definition/Background Information

Groundwater in the City has been found to contain PAH as a result of activities at a coal-tar distillation and wood preserving plant (Site) operated from 1917 to 1972. Numerous previous studies have identified PAHs in various aquifers beneath St. Louis Park and adjacent communities. Accordingly, the site of the plant operations was placed on the National Priorities List and the federal and state governments sought remediation of environmental contamination via United States District Court Case No. Civil 4-80-469. A more detailed explanation of site background is contained on Pages 3 through 9 of the Consent Decree. The City's consulting company is ENSR. ENSR works with the City to address issues concerning the Consent Decree-Remedial Action Plan (CD-RAP) which includes work plan development and implementation for various tasks, groundwater sampling, and compliance to the CD-RAP.

A summary of the aquifers which underlie the former wood preserving plant site, their approximate location below the surface level, the general use of the aquifers, and the relative maximum historical PAH concentration measured in each unit (as indicated by historical records and the federal government's Record of Decision in Case No. Civil 4-80-469) are as follows:

Aquifer	Approximate Depth (ft.)	Use	Approximate Upper Concentration of Total PAH
Drift-Platteville	0 - 90	Industrial/Monitor wells	1,000 µg/l off site
St. Peter	90 - 200	Municipal/Private drinking water wells	10 µg/l off site
Prairie du Chien-Jordan	250 - 500	Municipal drinking water wells	10 µg/l off site
Ironton-Galesville	700 - 750	Industrial	5 µg/l on site
Mt. Simon-Hinckley	800 – 1,100	Municipal drinking water wells	0.080 µg/l off site

More extensive information relative to the identified level of PAHs in the various aquifers is provided in the following reports:

- Annual Monitoring Reports for 1988 through 2000

- St. Peter Aquifer Remedial Investigation Report (March 30, 1989)

- Drift-Platteville Aquifer (Northern Area) Remedial Investigation Report (March 30, 1989)

The monitoring well locations for each aquifer are illustrated in Figures A-2 through A-6.

A.5 Project/Task Description and Schedule

A.5.A Tasks 1 through 8

The EPA, MPCA, MDH, the City, and Reilly have agreed to acceptable drinking water quality criteria for PAH.

Table A-1 lists the nominal reporting limits for the target compounds listed in the CD-RAP. Currently, only STL has conducted laboratory analyses of groundwater samples.

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from City wells (identified - SLP) 10 and 15. Further provisions of the RAP call for long-term monitoring of the influent and effluent of the GAC treatment system and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH in the groundwater and compare the analytical data with water quality criteria established in the CD-RAP. Currently, both the City and ENSR are collecting the groundwater samples. Typically, the City collects water samples from pumping wells (i.e. City owned wells) and ENSR collects water samples from non-pumping wells (i.e. monitoring wells). The specific objectives of the sampling and analysis program, and therefore, the intended end use of the data varies slightly for the different aquifers (Mt. Simon-Hinckley, Ironton-Galesville, Prairie du Chien-Jordan, St. Peter, and Drift-Platteville) being monitored in accordance with the CD-RAP.

If any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23 (CD-RAP Section 6.2.1), then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the wells from PAH resulting from the activities of Reilly at the Site. The analytical data would be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the CD-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking water wells, private or industrial wells, and monitoring wells are to (CD-RAP Section 7.3): 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control system, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the CD-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23).

In addition to water quality data generation, water level data will be used for the purpose of determining groundwater flow patterns in the Prairie du Chien-Jordan Aquifer.

The objectives of monitoring St. Peter Aquifer wells are to (CD-RAP Section 8.1.3): 1) monitor the distribution of PAH in the aquifer, thus evaluating a gradient control system installed at W410 in 1990, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site.

Upon its receipt, analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data, to drinking water cessation criteria for well W410, and to drinking water criteria established in the CD-RAP. Water level data will be used to evaluate groundwater patterns in the St. Peter Aquifer.

The objective of monitoring the Drift-Platteville Aquifer wells (CD-RAP Section 9.6) is to monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control systems. Groundwater analytical data will be used to compare levels of PAH in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the CD-RAP for PAH. Water level data will be used to evaluate groundwater flow patterns in the Drift-Platteville Aquifer.

In addition to the objectives for laboratory analytical data described above, field measurement data will be collected to aid in the groundwater sampling procedure. In accordance with MPCA Guidelines (January 1995), field measurements of temperature, pH, and specific conductance will be made for the purpose of determining that a sufficient volume of water has been purged from the well prior to sampling. The objective of those field measurements is to determine when three successive well volumes exhibiting equivalent temperature pH, and specific conductance have been purged from each monitoring well, so that representative samples may be collected.

A.5.B Project Schedule

The time period covered by this QAPP is from January 1, 2002, or the date of its acceptance and approval by the Agencies, whichever is later, to December 31, 2002. The QAPP is submitted annually by October 31st for the following year. The Annual Monitoring Report is submitted by March 15th for the previous year. The Site Management Plan outlines the sampling schedule and sampling locations for each year. The Consent Decree was signed in September 1986; therefore, this year marks the 15th year of the 30-year Consent Decree.

A.6 Quality Objectives and Criteria for Measurement Data

A.6.A Data Quality Objective

Step 1 – State the Problem. Groundwater in five aquifers in the City of St. Louis Park still contains PAH. The source controls at the Reilly site are designed to limit the spread of the PAH plume and drinking water treatment is provided to ensure an adequate supply of drinking water. The concentration of PAH needs to monitored continually to ensure that the public water supply meets the PAH criteria outlined in the CD-RAP and that the PAH concentrations in groundwater are not increasing.

Step 2 – Identify the decision. Groundwater and treated drinking water samples will be periodically collected and analyzed for PAH. The decision to made based on the data generated during these activities is whether the drinking water meets the PAH criteria and whether PAH is spreading in the aquifers.

Step 3 – Identify Inputs to the Decision. The groundwater monitoring network in St. Louis Park is well established based on monitoring since 1987. A series of wells in each aquifer as discussed in section A.6 are to provide sampled data for this program. The samples will be analyzed for PAH to evaluate contaminant concentrations.

Step 4 – Define the Boundaries of the Study. The spatial boundaries of the study are based on the plume geometry presented in the Annual Monitoring Reports. The spacial boundary varies between aquifers, however, the plume is generally contained within the City of St. Louis Park city limits.

Step 5 – Develop a Decision Rule. If municipal drinking water sample exceed the Advisory Level for PAH, then it will be resampled according to the procedures described in Section 12 of the CD-RAP. With the exception of the above scenario, groundwater sampling frequency is defined in Tables B-2 and B-3.

Step 6 – Specify Limits on Decision Errors. The only limitations are the data quality limitations based on the analytical methodologies used.

Step 7 – Optimize the Design for Obtaining Data. The wells to be sampled for this program were selected to be representative of each of the five aquifers being monitored. The sampling design has been developed to support the objective of comparing PAH concentrations to drinking water criteria and historic water quality results.

ENSR works with the City to address issues concerning the CD-RAP that includes work plan development and implementation for various tasks, groundwater sampling, and compliance to the CD-RAP.

For active drinking water wells, STL will notify the City by telephone, within 24 hours of completing an analysis, whenever a sample analysis is shown to exceed the following Advisory Levels or Drinking Water Criterion:

Parameter	Advisory Level	Drinking Water Criterion
Sum of Benzo(a)pyrene and Dibenz(a,h)anthracene[1]	3.0 ng/L[1]	5.6 ng/L
Total Carcinogenic PAH[2]	15 ng/L[3]	28 ng/L[3]
Total Other PAH	175 ng/L	280 ng/L

1 Or the detection limit, whichever is largest

2 See Table A-2

3 Different concentrations for additional carcinogenic PAH may be established in accordance with the procedure specified in Part D.1 of the Consent Decree

A.6.B Measurement Performance Criteria - PARCC

The principal objectives of the QAPP pertain to the collection of data that are sufficient to monitor the effectiveness of the GAC treatment system and to detect changes in groundwater quality. Therefore, the quality of the data gathered in this project can be defined in terms of the following elements:

- Precision – Precision is the degree of agreement among repeated measurements of the same characteristic (analyte, parameter, etc.) under the same or similar conditions. Precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits.

  Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per 10 analytical samples (ten percent duplication), or Sample Delivery Group (SDG), whichever is less. Precision will be measured through the calculation of relative percent difference (RPD). The RPD is calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} x\,100$$

where: RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (duplicate)

Precision in the laboratory is assessed through the calculation of RPD for duplicate samples, either as matrix spike/matrix spike duplicates (MS/MSDs) or as laboratory duplicates, depending on the method. Precision control limits for laboratory analyses are discussed in Table A-3.

- Accuracy – Accuracy is the extent of agreement between an observed value (sample result) and the accepted, or true, value of the parameter being measured. The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Laboratory and method accuracy are calculated as a percentage using the following equation:

$$Accuracy = \frac{X}{T} x\,100$$

where: X = the observed value of measurement
T = "true" value

Laboratory accuracy is assessed through the analysis of MS/MSDs, laboratory control samples (LCSs), and surrogate compounds, and the subsequent determination of percent recoveries (%Rs). Accuracy control limits are given in Table A-3. The spike recovery is calculated in percentage using the following equation:

$$\%\mathrm{Recovery\_Accuracy} = \frac{SS - US}{C} x\,100$$

where: SS = Spiked Sample Concentration
US = Unspiked Sample Concentration
C = Spike Concentration added

- Representativeness – Representativeness is the extent to which reported analytical results truly depict the PAH concentration in the sampled environment. Representativeness is optimized through proper selection of sampling sites, times and procedures, through proper sample preservation, and through prompt extraction and analysis.

  It is essential that representative groundwater samples be retrieved for laboratory analyses. Accuracy and precision in the measurement of parameters used to monitor groundwater as it is purged from monitor wells and piezometers will be achieved through the use of standard monitoring procedures carried out continuously during the sample retrieval task. Field measurement equipment will be calibrated in accordance with the manufacturer's recommendations, as outlined in Table B-4, and appropriate corrective action will be initiated whenever measured accuracy and precision do not meet pre-established limits. Precision and accuracy of field measurement devices will be tested by taking duplicate samples and calculating the relative percent difference using the formula presented above. Duplicate field readings will be completed at a 10 percent frequency.

- Comparability – Comparability is the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures outlined in the SOPs in Appendices A and B.

- Completeness – Completeness is assuring that a sufficient number of successful (valid) measurements have been collected to characterize the concentrations of PAH in the influent and effluent of the treatment system and in the aquifers of interest over a period of time. For this project, the completeness objective is that 95 percent of the laboratory analyses and 95 percent of the field measurements will produce valid data. Field data will be supplemented by resampling if necessary to ensure completeness.

- Sensitivity – Sensitivity is the ability of the method or instrument to detect the contaminant of concern at the level of interest. Determination of instrument sensitivity is accomplished by calibration using multiple concentrations of the analytes of interest. Once instrument sensitivity is demonstrated, analysis of replicate spiked samples of deionized reagent water at a concentration of one to five times the instrument sensitivity, is used to determine method sensitivity (i.e. method detection limit).

- Traceability - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

The fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment and correction, as follows:

1. Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection and training of skilled, qualified personnel.

2. Quality assessment through a program of regular audits and inspections to supplement continual informal review (refer to Section C.1 of this QAPP).

3. Permanent correction of conditions adverse to quality through a closed-loop corrective action system.

The City sampling program QAPP has been prepared in direct response to these goals. The QAPP describes the quality assurance program to be implemented and the quality control procedures to be followed by STL during the course of laboratory analyses in support of the various site investigation studies for the City Site. The Quality Assurance objectives will include field blanks, method blanks, field duplicates, surrogate spikes, matrix spikes and matrix spike duplicates. Precision, accuracy and completeness criteria are established for each parameter of interest. The specific criteria for each analysis and parameter are set forth in detail in the following sections:

Objective	Frequency (%)	Sections Discussing Criteria
Field Duplicates	10	B.5.A
Field Blanks	10	B.5.A
Method Blanks	5	B.5.B
Surrogate Spikes	100 of GC/MS analyses	B.5.B
Matrix Spikes/Duplicates	5*	B.5.B
* One per group of 20 or fewer investigative samples		

A.7 Special Training Requirements/Certification

All ENSR and City personnel working on the project will be properly trained, qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- Organization and lines of communication and authority
- Overview of the Site Management Plan and QAPP
- Documentation requirements
- Decontamination requirements
- Health and Safety considerations

Training of field personnel will be provided by the Field Coordinator or a qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency in the analytical methods to be employed. All analysts must demonstrate their ability to perform any analytical SOP before they are allowed to analyze project samples. This is demonstrated through the successful analysis of two consecutive sets of proficiency QC samples. Each set of proficiency samples consists of two spiked aliquots of a control mix and one method blank, the spike concentrations and acceptance limits are the same as for the LCSs described in the SOP. All proficiency data must be reviewed by the supervisor and a representative of the QA office. The QA manager submits to the department supervisor a written report or certificate of proficiency indicating either need for corrective action or acceptable performance. The proficiency certificate is included in the employee's training file.

A.8 Documentation and Records

A.8.A Documents and records that will be generated

Control of, and accounting for documents generated during the course of the project is achieved by assigning the responsibility for document issuance and archiving. Table A-4 lists the key documentation media for the project and corresponding responsible parties for issuance, execution and archiving.

A.8.B Data Reporting Package Format and Documentation Control

STL shall prepare summary reports and data packages in standard STL format. The reporting forms will show the target lists of parameters, surrogates and spiking compounds for the Low-Level PAH.

STL has determined the method detection limits for the ppt PAH analysis of water samples, utilizing GC/MS selected ion monitoring, according to the method described in Appendix B to Part 136 of the Friday, October 26, 1984, Federal Register, Vol. 49, No. 209 - Definition and Procedure for the Determination of the Method Detection - Revision 11.1. Table A-5 lists the compounds, the observed concentrations of seven replicates spiked at five ppt, the standard deviations and the method detection limits.

These calculated method detection limits will be used in sample reporting as follows:

- Analytes detected at concentrations greater than or equal to the calculated method detection limits will be reported with no qualifiers

- Analytes that are not detected will be reported as (non-detected) ND

- Analytes that are detected at concentrations less than the reporting limit but greater than the calculated method detection limits will be reported followed by a "J" qualifier which is used in the EPA CLP to indicate that a reported value is below the sample quantitation limit and above the method detection limit

The various items in the data package are listed below:

- **Sample Data Summary Report:**

  Report Cover
  Table of Contents
  Case narrative
  Tabulated target compound results by fraction
  Surrogate spike analysis results by fraction
  Blank data by fraction
  LCS/LCSD results by fraction
  Matrix spike/matrix spike duplicate results by fraction
  Chain of Custody Record

- **Sample Data Package:**

  Raw data

The City will present reports in a manner consistent with the requirements of Section 3.1 of the RAP. In addition, data packages containing all elements listed above will be presented for the sample analyses completed, if so directed by the EPA. The EPA shall be responsible for identifying the specific sample analyses for which data packages will be provided.

A.8.C Data Reporting Package Archiving and Retrieval

The final evidence (or data) files will be maintained for the 30-year period specified in the RAP. Evidence files will consist of (at a minimum): all project reports, reporting limits, chain-of-custody documentation, quality control data for blanks and matrix spikes, results forms, nonconformance documentation and a file custodian. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues, is included in the package. The City will maintain these files in a secure, limited access area, under the custody of the Project Manager. STL maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. This data will be available upon request.

B. DATA GENERATION AND ACQUISITION

B.1 Sampling Process Design

Samples will be collected by ENSR and City personnel in accordance with the1995 MPCA Groundwater Sampling Guidance Document. This element describes where the groundwater samples will be taken, the number of samples to be taken and the sampling frequency. In addition, this element discusses general QAPP provisions relevant to sample collection, containerization, packaging and shipping activities (SOPs 7130 and 7510; Appendix A).

The Site Management Plan outlines the scope of work to be performed in order to monitor the groundwater in the St. Louis Park, Minnesota, area in accordance with the CD-RAP and the Agencies' October 3 and 19, 2000, letters related to the Reilly N.P.L. Site. Included in this Plan are: 1) the identity of wells to be monitored, 2) the schedule for groundwater monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting. Although a GAC treatment system has been constructed to treat water from well W23 and the Drift-Platteville Aquifer source control wells prior to its discharge to surface water receivers, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the CD-RAP. Similarly, a GAC treatment system has been constructed to treat water from well SLP4 prior to discharge to the municipal water supply system; however, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the CD-RAP.

The overall sampling program is summarized in Tables B-1, B-2, and B-3, and Figures A-2 through A-6.

B.2 Sampling Methods Requirements

This element describes how the groundwater samples will be collected. In addition to proper sample collection, preservation, storage and handling, appropriate sample identification procedures and chain of custody are necessary to help insure the validity of the data.

B.2.A Sampling SOPs

Groundwater samples will be collected in accordance with ENSR SOP 7120 found in Appendix A.

B.2.A.1 Groundwater Sampling and Water Level Measurements

Groundwater samples will be collected and water levels measured in accordance with the procedures outlined in this QAPP. The wells involved in the monitoring program include municipal and commercial wells, piezometers and groundwater monitoring wells (Table B-3). Sampling procedures to accommodate the dimensions and configuration of each type of well are described below. Further details on well dimensions, water level measurements and sample acquisition strategies are given in the Site Management Plan.

The importance of proper sampling of wells cannot be over emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the groundwater at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure. Sample collection will always proceed from the less contaminated sampling points to the monitoring points containing progressively higher concentration of PAH.

B.2.A.2 Sample Collection - Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in purging the monitoring wells and piezometers, flexibility in the approach to the method of well purging is necessary. This QAPP proposes that the sampling team be given latitude in the selection of purge equipment necessary to complete the task (various pumping equipment and procedures that may be used for purging monitoring wells are described in SOP 7130 and MPCA's 1995 Guidelines). In all cases where no dedicated pump exists, samples will be retrieved using laboratory-cleaned, stainless steel or Teflon bailers as described below.

Table B-3 specifies that Prairie du Chien-Jordan Aquifer monitor well W70, and St. Peter Aquifer monitor wells W24 and W33 be sampled. These wells are equipped with a dedicated submersible pump and it will be the responsibility of the sampling team to determine if the pump is operable. In the event the dedicated pump within any individual well is operable, well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with monitoring parameters established herein. In the event the dedicated pump within any individual well is inoperable, the pump will be removed, if possible, and purging/sampling procedures will be as established below

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a non-dedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH and specific conductance of the purge water will be monitored using a Horiba water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or Teflon bailer and a new length of nylon or polyester rope. All non-dedicated purging and sampling equipment will be decontaminated before use and between sampling points as described in Section B.2.B. An equipment blank will be collected at the frequency of one for each 10 samples collected from wells that have non-dedicated sampling equipment.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without pre-rinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

B.2.A.3 Sample Collection - Pumping Wells

At active pumping wells, the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. A clean electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered.

If it cannot be determined that a well has been pumping at some time during the 24-hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

B.2.A.4 Sampling Procedures - GAC Treatment System

Sampling points will be flushed for at least five minutes before collecting a sample. Each PAH sample and matrix spike sample will be collected in six 1-liter amber glass bottles, which should be filled and capped in succession. PAH sample bottles will not be rinsed before being filled.

The GAC treated water samples will have to be collected from two sample taps, one for each column (see Figure B-1). This will be done by filling three 1-liter bottle from the first column sample tap and then three more bottles from the second (three from each for duplicate samples). No notations distinguishing the two taps will be made on the labels.

Field blank samples will be prepared by transferring contaminant-free deionized water provided by STL into sample bottles in a fashion as closely similar to actual sample collection as possible. Field blank sample bottles will be filled and capped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area in which GAC treated water samples are collected.

Field duplicate and matrix spike duplicate samples will be obtained by filling six 1-liter bottles at the sampling point by the procedure described above and assigning a different sample number to each of the resulting two bottle samples.

Chain-of –custody forms will be completed and all samples collected for this program will be packed, cooled to a temperature less than 4°C, and shipped overnight to STL on the day they are collected.

The sampling team must recognize that great care is required to collect samples for part per trillion level PAH analyses that are free from outside contamination. PAH compounds are present in cigarette smoke, engine exhaust and many petroleum derived oils, among other sources. There will be no smoking anywhere in the GAC treatment building for at least 72 hours prior to the day on which PAH samples are to be collected. Similarly, no vehicles will enter the GAC treatment building and the large access door will stay closed for at least 72 hours prior to the sampling day. Disposable gloves will be worn when collecting, handling and packaging samples. Sample bottles will remain in closed shipping coolers until they are needed, and will be packaged and sealed for shipment as soon as possible after sampling.

B.2.B Cleaning and Decontamination of Equipment/Sample Containers

The field decontamination procedure to be used on sampling equipment, which comes into contact with groundwater samples, is as follows:

- Disassemble equipment, if applicable
- Wash with Alconox and potable water
- Rinse with potable water

Sampling equipment, such as pumps, are decontaminated prior to sampling each well. The effectiveness of the decontamination procedures is measured by collecting and analyzing equipment blank samples.

The laboratory decontamination procedure to be used on sampling equipment, such as bailers, which comes into contact with groundwater samples, is as follows:

- Disassemble equipment
- Rinse with methanol
- Scrub with hot soapy water
- Rinse three times with hot deionized water
- Set on aluminum foil, dull side up, air dry
- Bake for one hour at 200°C
- Wrap with aluminum foil, dull side in

Pre-cleaned 1-liter amber bottles are supplied by STL for sample containerization

B.2.C Field Equipment Maintenance, Testing and Inspection Requirements

All field measurement equipment will be controlled to ensure that measurements obtained are accurate and defensible. Table B-4 summarizes the parameters to be monitored including calibration and frequency, and quality control criteria (also refer to Appendix A, SOPs 7121, 7123, and 7124).

In addition, these measurement devices will be issued through a formal equipment tracking system and operated by trained personnel.

B.2.D Inspection and Acceptance Requirements for Supplies/Sample Containers

This section documents the activities that will be performed to ensure that all sampling supplies and containers are free of contaminants of concern. For PAH, 1-liter amber glass bottles will be used (Table B-5). Caps will be fitted with pre-cleaned Teflon liners. Six bottles are required for each Low-

Level PAH sample collected and two bottles for each 8270C PAH and Extended Analysis sample collected. An independent commercial firm shall provide pre-cleaned bottles to STL for use on this project.

In the event STL is required to prepare bottles for sampling, the bottles will be prepared as follows:

1. Wash bottles with hot detergent water.

2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.

3. Rinse with Burdick & Jackson quality redistilled acetone, followed by equivalent quality methylene chloride.

4. Allow to air dry in a contaminant-free area.

5. Caps and liners must be washed and rinsed also. Bottles should be stored and shipped with the Teflon-lined caps securely fastened.

6. Vendor certified clean sample bottles can be used in place of above cleaning procedures.

STL provides adequate sampling supplies (coolers, containers, packaging supplies, etc.) that are stored by the City at Water Treatment Plan Number 1. Supplies are ordered as needed, by the Field Sampling Team members. STL is responsible for ensuring the cleanliness of the sampling supplies, and the Field Sampling Team is responsible for using only clean containers and supplies.

B.3 Sample Handling and Custody Requirements

This element of the QAPP insures that:

- Samples are collected, transferred, stored, and analyzed by authorized personnel;
- Sample integrity is maintained during all phases of sample handling and analysis; and
- An accurate written record is maintained of sample handling and treatment from the time of collection through laboratory procedures to disposal.

B.3.A Sampling Handling

The St. Louis Park Groundwater Study is a cooperative effort between the City and ENSR, whose responsibilities include sample retrieval, and STL, whose responsibilities include sample analysis. Proper sample handling and analysis is essential to the success of the study, therefore a formal sample custody procedure has been developed to insure the integrity of all samples.

Sample labels shall be completed for each sample using waterproof ink. The information recorded on the sample label includes:

Sample Number - Unique coded sample identification number as described below.

Date and Time – The date of collection and the 4-digit number indicating the military time of collection.

Sampler - Initials of person collecting the sample.

Remarks - Any pertinent observations or further sample description. The sample number includes three parts (source code, sampling point code, and date code) in the following sequence:

XXX-YYYYY-ZZZZZZ

XXX	=	Source Code	
		GAC Treatment System	= GAC
		Mt. Simon-Hinckley Aquifer	= MSH
		Ironton-Galesville Aquifer	= IGV
		Prairie du Chien-Jordan Aquifer	= PCJ
		St. Peter Aquifer	= STP
		Drift-Platteville Aquifer	= DPV
		Groundwater Treatment Facility	= GTF
YYYYY	=	Sampling Point Code (ex: SLP6 = Monitoring Well SLP6)	
ZZZZZZ	=	Date Code (ex: 053099 = May 30, 1999)	

Those samples which will be taken in accordance with this QAPP for quality control purposes will be identified by appending to the sampling point codes the following:

Field blank = FB
Field duplicate = D
Matrix spike = MS
Matrix spike duplicate = MSD

As an example, a field blank sample taken for the Mt. Simon-Hinckley Aquifer, sampling point SLP11 on January 1, 1991, would be identified as follows:

MSH-SLP11FB-010191

During the sampling event, one sample will be taken per sampling point unless it is duplicated. Duplicate samples will be collected as specified in Table B-1. Those samples collected for matrix spike analysis will be selected at the time of sampling and labeled in the field.

After collection, identification, and preservation, the sample will be maintained under chain-of-custody procedures discussed below.

Packaging and shipment of samples shall be in accordance with SOP 7510 (Appendix A). The samples will be iced or refrigerated at 4°C from the time of collection until extraction. PAHs are known to be light sensitive; therefore, samples will be stored in amber bottles and kept away from prolonged exposure to light. All samples for gas chromatography mass spectrometry (GC/MS) analysis will be extracted within seven days of collection. The analysis will be completed within 40 days following extraction.

Samples will be protected from breakage and shipped in coolers at a temperature of 4°C ± 2°C. An overnight carrier will be selected to insure delivery at the laboratory within 24 to 36 hours after collection. Samples received at the laboratory will be checked for leakage and a notation made regarding sample temperature at time of receipt. All samples should be stored in an organic-free refrigerator at 4°C ± 2°C.

Table B-5 outlines the required sample volumes, container types, number of containers, preservation procedures, and holding time for each analytical parameter.

B.3.B Sample Custody

To maintain and document sample possession, chain-of-custody procedures will be followed. A sample is under custody if:

- It is in someone's possession
- It is in someone's view, after being in their possession
- It is in a designated secure area

Samples are accompanied by a Chain-of-Custody Record. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst at the laboratory.

Minimum information recorded on the chain-of-custody record, in addition to the signatures and dates of all custodians, will include:

- Sampling site identification
- Sampling date and time
- Identification of sample collector
- Sample identification
- Sample description (type and quantity)
- Analyses to be performed

Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. Shipping containers will be sealed for shipment to the laboratory. The method of shipment, courier name(s) and other pertinent information are entered in the "Remarks" box. The last copy of the form is retained by the field sampling team and the original and remaining copies of the custody record are placed in the container. After the container is closed, place the custody seals on the container.

Whenever samples are split with another laboratory, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

B.3.B.1 Field Custody Procedures

In addition to sample labels and chain-of-custody forms, a field notebook will be maintained by the sample team leader to provide a daily record of significant events. Information to be documented in the notebook will be groundwater sample collection records, calibration records, list of samples collected and any other pertinent information such as weather conditions, site visitors, ease/difficulty of retrieving samples, etc. All entries will be signed and dated. All members of the sampling team will use this notebook. The notebook will be kept as a permanent record.

Samples will be labeled in accordance to the procedure outlined in section B.3.A. Samples will be protected from breakage and shipped in coolers at a temperature of 4°C ± 2°C. An overnight carrier will be selected to insure delivery at the laboratory within 24 to 36 hours after collection. Packaging and shipment of samples shall be in accordance with SOP 7510 (Appendix A).

B.3.B.2 Lab Custody Procedures

Samples entering the STL analytical facilities located in Arvada, Colorado proceed through an orderly chain-of-custody sequence specifically designed to insure continuous integrity of both the sample and documentation.

Appendix B contains SOPs that address the aspects of facility security and sample custody. Figure B-2 shows the data collection process flow chart.

B.3.B.3 Final Evidence Files

The final evidence (or data) files will be maintained for the period specified in the RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of (at a minimum): all field documents, logs, project reports, raw data, continuing calibration checks, detection limits, chain-of-custody documentation, quality control data for blanks and matrix spikes, results forms, nonconformance documentation and a file custodian. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues, is included in the package. The City will maintain these files in a secure, limited access area, under the custody of the Project Manager. STL maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. This data will be available upon request

B.4 Analytical Methods Requirements

As specified in the Consent Decree, three types of analyses are to be performed as part of the RAP for this project. Table B-6 outlines the three types of analyses and the details of the specific analytical procedures are presented in subsequent subsections.

B.4.A Low-Level Analysis for PAH

Low-Level refers to the determination of a specific list of 31 PAHs using GC/MS with operation in the selected ion monitoring (SIM) mode. The list of target PAH compounds contains carcinogenic and non-carcinogenic compounds and is shown in Table B-7 of the QAPP. The list includes 15 compounds that are not on EPA's priority pollutant, Appendix IX or Superfund target compound list. The analytical methodology is based on well known principles of GC/MS technology. Although there is no EPA method that embodies this technique for this class of compounds, methods developed for the measurement of polychlorinated dibenzodioxins (e.g., Methods 613 and 8280) are based on selected ion monitoring technology.

A method has been developed for the analysis of selected target PAH and heterocycle compounds at the part per trillion level (ppt, ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by GC/MS in the SIM mode. The method is generally applicable for the measurement of any PAH or related compound. For this project, only those compounds listed in Table B-7 will be determined.

In summary, a measured volume of sample is extracted with methylene chloride. Analysis of the concentrated extract is performed by GC/MS using the selected ion monitoring scanning mode under electron impact ionization conditions. Specific details of this methodology can be found in Appendix B, Polynuclear Aromatic Hydrocarbons by Selective ion Monitoring for City of St. Louis Park. This method is designed to analyze samples containing up to 600 ppt of an individual PAH. With dilution of the sample extract, the effective range of the method can be extended into the ppb range. However, sample dilutions may result in loss of information concerning recovery of surrogates. For this reason, an optional sample preparation technique is contained in the method. This optional technique can be used if historical information indicates that the target compounds are present in concentrations in excess of 600 ppt.

B.4.B Full Scan 8270C Analysis for PAH

This analysis uses the methodology contained in SOP No. CORP-MS-0001DEN; "GC/MS Analysis Based on Method 8270C and 625" (Appendix B). The 16 target compounds for method SW846 8270C are listed in Table B-8. The full scan method is used for analyzing PAH concentrations in the parts per billion range and is also referred to as the PAH (ppb) method.

B.4.C Extended Analysis

Some samples are analyzed for the specific list of compounds shown in Table B-9 of the QAPP using scanning GC/MS. This list, termed "Extended" analyses, includes additional PAH, specific acid (phenolic) compounds and a provision for "identifying" unknown compounds. Unknown compounds will be identified and reported from the analysis of the acid fraction only. The extended analyses are performed using SW-846 protocols with the appropriate modifications.

The target compounds listed in Table B-9 are measured using the methodology contained in SW-846, method 8270C for semivolatile organics. The only deviations from this method are as follows:

1. The calibration is performed as described in Section B.7 of the QAPP
2. The only target compound in the analytical reports are those listed in Table B-9

B.4.D Turnaround Time

In accordance with Section 3.2 of the RAP, STL has agreed to a 30-working day turnaround. The City, however, makes no enforceable commitment under the RAP except for a maximum of seven days from time of sample collection for extraction of organics and 40 days following extraction for analysis of organics. For non-organic analyses, the City makes no enforceable commitment under the RAP except to meet the recommended maximum analytical holding times.

B.5 Quality Control Requirements

Quality control (QC) is the overall system of technical activities that measure the attributes and performance of a process, item or service against defined standards to verify that they meet the stated requirements. Acceptable limits of performance are defined for each QC check and sample used in the project.

B.5.A Field Sampling Quality Control

Field blank samples will be prepared by transferring contaminant-free deionized water, provided by STL, into sample bottles in a fashion as closely similar to actual sample collection as possible. This will involve collecting samples through any non-dedicated sample equipment that is decontaminated between samples. Field blank sample bottles will be filled and capped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area where samples are being collected at a rate of one per day or where more than 10 samples are collected in a day at a rate of one field blank per 10 samples.

Duplicate samples will be collected by alternately filling sample bottles from the source being sampled. For example for six liter sample collections, one bottle will be filled for the sample, then one bottle for the duplicate, then a second bottle for the sample and then a second bottle for the duplicate. Duplicates will be taken for each analysis type and each sample type, at a rate of one duplicate sample being collected for each 10 samples, with a minimum of one duplicate for any sample batch. There are two sample types for this program: GAC treatment system water and groundwater.

B.5.B Analytical Quality Control Checks

The internal quality control checks will include field blanks, method blanks, surrogate spikes, duplicate analyses, monitoring of internal standard area, and matrix spike analyses. Each quality control check has a specific level of performance that will be reevaluated in an ongoing basis and amended as appropriate through mutual agreement of the EPA, MPCA, and City. The QC check for lab analysis are summarized in Table B-10 and the specific details are presented below.

B.5.B.1 Low-Level and Full Scan 8270C PAH Analyses

Internal quality control checks for the Low-Level and Full Scan 8270C PAH analyses will consist of method blanks analysis, surrogate compound analysis, matrix spike analysis, analysis of duplicate samples, and monitoring of internal standard areas.

B.5.B.1.1 Method Blank Analysis

A method blank consists of deionized, distilled laboratory water carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank volume must be approximately equal to the sample volumes being processed.

Method blank analyses are performed at the rate of one per case[1], each 14-calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix, or whenever samples are extracted by the same procedure, whichever is most frequent.

Different control limits have been established relative to method blanks for the Low-Level and Full Scan 8270C analyses since the target compounds in Table B-7 are present as "laboratory contaminants" in method blanks at the ppt concentration level.

For the Low-Level analyses, an acceptable method blank analysis must not contain any carcinogenic PAH in Table B-7 at concentrations greater than or equal to the Method Detection Limits (MDL) in Table A-5 or any non-carcinogenic PAH at a concentration greater than five times the MDL. The criteria for an acceptable 8270C method blank are out lined in the SOP in Appendix B. If the method blanks do not meet criteria, the analytical system is out of control and the source of the contamination must be investigated and corrective measures taken and documented before further sample analysis proceeds.

B.5.B.1.2 Surrogate Compound Analysis

As detailed in the STL SOP (Appendix B), the laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compound will be spiked into the sample prior to extraction to measure individual sample matrix effects associated with sample preparation and analysis.

STL will take corrective action whenever the surrogate recovery is outside the acceptance criteria shown below. The corrective action is described in Section C.1.B of this QAPP.

In addition, if the recovery of any surrogate is less than 30 percent, the narrative will list the sample together with a comment concerning a possible low bias to the sample result.

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[1] A case is a group or a set of samples collected from a particular site over a given period of time.

Surrogate	Acceptance Criteria Percent PAH PPT
Naphthalene-d8	21 - 108
Fluorene-d10	41 - 162
Chrysene-d12	10 - 118

B.5.B.1.3 Matrix Spike/Matrix Spike Duplicate Analysis

Low-Level PAH matrix spike and matrix spike duplicate samples will be analyzed as outlined in the STL SOP DEN-MS-0005, "PAHs by Selective Ion Monitoring for City of St. Louis Park" (Appendix B). Full Scan 8270C PAH matrix spike and matrix spike duplicate samples will be analyzed pursuant to applicable criteria in STL SOP CORP-MS-0001DEN.

The laboratory will spike and analyze five percent matrix spike and matrix spike duplicate samples. STL will spike seven representative compounds into water. These compounds and the spiking levels are listed below:

Compound	Low-Level PAH (ng/L)
Naphthalene	10
Fluorene	10
Chrysene	10
Indene	10
Quinoline	10
Benzo(e)pyrene	10
2-methylnaphthalene	10

The matrix spike criteria for data validity are as follows:

- The Matrix Spike - Matrix Spike Duplicate **average** for each spike compound must fall between the established acceptable limits.

Matrix Spike Limits

Compound	Low-Level
Naphthalene	20 – 150
Fluorene	20 – 132
Chrysene	20 - 132
IH-Indene	20 - 150
Quinoline	20 - 150
Benzo(e)pyrene	20 - 150
2-methylnaphthalene	20 - 150

- Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:

  1. 10 percent for chrysene and benzo(e)pyrene
  2. 20 percent for all other compounds

Corrective action will be performed if these criteria are not achieved as described in Section C.1.B.

Spiking limits and acceptable percent recovery for method 8270C are listed in SOP CORP-MS-0001DEN (Appendix B).

B.5.B.1.4 Duplicates

Relative percent difference between duplicates will be calculated for each detected compound per procedures outlined in Section A.7.B of this QAPP.

B.5.B.1.5 Internal Standard Areas

The area of the internal standard will be monitored on each analysis. The area from the daily calibration standard will be used to set a daily acceptance criteria. If the internal standard areas in samples changes by more than a factor of two (-50 percent to +100 percent) from the daily standard, corrective action must be performed. Additionally, the retention times of internal standards must agree to +/-30 seconds of the daily standards.

B.5.B.2 Extended Analysis

The internal quality control checks for Extended Analyses will consist of surrogate spikes, matrix spikes, matrix spike duplicates, method blanks, etc.

B.6 Instruments/Equipment Testing, Inspection, and Maintenance Requirements

This section describes the procedures used to verify that all instruments and equipment are maintained in sound operating condition and in working order when needed.

B.6.A Field Instrument Maintenance

All field equipment shall be inspected daily for damaged or missing pieces, which will be replaced as needed.

B.6.A.1 Thermometer

The field worker will handle the thermometer with care to preserve its measurement integrity. After each use, the thermometer will be rinsed with de-ionized or potable water, wiped dry, and returned to its protective case.

B.6.A.2 Water Level Measurement Tape

Before each use, the battery will be checked using the equipment's element test function, and replaced if necessary. The tape and probe will be wiped clean and rinsed with de-ionized or potable water after each use.

B.6.A.3 Horiba U-10

The Horiba U-10 shall be maintained in accordance with the manufacturer's requirements. In particular, the battery will be checked daily, and replaced if necessary. The instrument shall be operated and stored at temperatures above freezing, to avoid damaging the instrument. After each use, the instrument will be rinsed with potable or de-ionized water, wiped dry and returned to its storage container.

B.6.B Laboratory Instrument Maintenance

Since instrumental methods of analysis require properly maintained and calibrated equipment, the operation and maintenance of modern analytical instrumentation is of primary importance in the production of acceptable data. The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system. The instrument mainentance schedules are summarized in Table B-11 and B-12.

Individual instrument logbooks are maintained for each piece of equipment and located near the instrument. General information contained in the logbooks include:

- Inventory information: Equipment name, model number, serial number, manufacturer, date of acquisition
- Service tasks and intervals: Cleaning, calibration, operation based on the manufacturer's recommended schedule, and previous laboratory experience
- Service record: Date of breakdown, date of return to service, downtime, problems, repairs, cost of repairs, who performed the repairs, parts required, etc.
- Calibration/performance checks
- Daily operational notes

Analysts are referred to manufacturers' operating manuals for specific procedures to be followed in the operation and/or maintenance of the individual instruments.

Within each laboratory, Group Leaders and analysts actually implement and document the maintenance performed.

Each instrument or piece of equipment shall be uniquely identified. Each operating unit shall maintain the following:

- Instrument/equipment inventory list
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit

The record of maintenance shall include at a minimum:

- Actions taken, including parts replaced
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative

STL documents and describes in detail instrument or equipment preventive maintenance in operation-specific SOPs. SOPs are specific to the type of instrument or equipment being used for sample analysis.

B.7 Instrument Calibration and Frequency

Calibration is required to ensure that field and laboratory analytical systems are operating correctly and functioning at the proper sensitivity to meet established detection limits. For this project, calibration is required for field measurements of temperature, pH, and specific conductance. Appendix A contains the SOPs that describes calibration procedures for field measurement instruments. This project also requires calibration for the three laboratory analyses (Low-Level, Full Scan 8270C, and Extended). These three analyses are defined in Section B.4 of this QAPP.

The laboratory is required to maintain logbooks that contain instrument usage, preventive maintenance, repairs, corrective actions, initial calibrations, daily calibration verifications and calibration standards used.

The specific calibration requirements for each of these analyses are summarized in the subsections below.

B.7.A Low-Level Analysis

The calibration requirements are described in detail in the SOP for ppt PAH analyses (Appendix B). The discussion below highlights the key aspects of the calibration requirements.

Prior to use of the method for Low-Level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of the analysis.

A midpoint calibration standard is analyzed at the start of each 12-hour calibration sequence and the area of the primary characteristic ion is tabulated against concentration for each compound. The response factor (RF) for each compound listed in Table B-7 is calculated.

These daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ±35 percent of the corresponding calibration curve value, the analysis may proceed. If, for any analyte, the daily response factor is not within ±35 percent of the corresponding calibration curve value, the system is out of control and corrective action must be performed.

The quantitation mass ion, which represents the 100 percent abundance ion, is selected for quantitation and for the daily response factor measurement. The second ion, or confirmation ion, is used for confirmation of the identification. The daily response factor for the quantitation mass ion is compared to the initial calibration curve. During the analysis of the daily calibration standard, the percent abundance of the confirmation ion is obtained. This percent abundance is used for identification purposes for samples analyzed during that day. The percent abundance values shown in Table B-8 are typical values.

Mass tuning will be performed using the mass calibration compound FC43. Tuning will be performed to maximize the sensitivity of the mass spectrometer for the mass range of compounds being analyzed.

Laboratory studies have shown that Benzo(j)fluoranthene will coelute with either Benzo(b)fluoranthene or Benzo(k)fluoranthene depending on the relative concentration of these two compounds in solution. Benzo(j)fluoranthene cannot be consistently separated by this method. Therefore, if present, it will be detected and reported as Benzo(b) and/or Benzo(k)fluoranthene.

B.7.B Full Scan 8270C Analysis for PAH

The calibration requirements are described in detail in the SOP CORP-MS-0001DEN "GC/MS Analysis Based On Methods 8270C and 625" in Appendix B. The discussion below highlights the key aspects of the calibration requirements.

Prior to use of the method, a five-point or greater response factor calibration curve must be established showing the linear range of the analysis. A midpoint calibration standard is analyzed at the start of each 12-hour calibration sequence and the area of the primary characteristic ion is tabulated against concentration for each compound. The response factor (RF) for each compound listed in Table B-8 is calculated.

These daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within +/- 30 percent of the corresponding calibration curve value, the analysis may proceed. If, for any analyte, the daily response factor is not within +/- 30 percent of the corresponding calibration curve value, the system is out of control and corrective action must be performed.

B.7.C Extended Analysis

In addition to the compounds listed in Table B-8, the compounds shown in Table B-9 are required to be determined in the extended monitoring program. This extended list of compounds include phenolics and other PAHs specified for this project.

Analyses for the extended list of compounds will be performed on the semivolatile extract prepared as described in SW846, method 8270C.

Example retention times, quantitation ions and the internal standards determined at the laboratory for 7,12-dimethylbenz(a)anthracene and 3-methylcholanthrene are listed in Table B-13.

C. ASSESSMENT/OVERSIGHT

This element details the procedures used to ensure proper implementation of the QAPP and used to assess of the effectiveness of the QAPP.

C.1 Assessment and Response Actions

This section identifies the number, frequency, and type of planned assessment activities that will be performed for the project. The ability of the Sampling Team to successfully monitor pumping wells and monitor wells, and the ability of the laboratory to successfully analyze groundwater samples will be confirmed by a series of assessments conducted in conjunction with the implementation of the groundwater monitoring program established in the CD-RAP. See Table C-1 for a summary of planned assessments.

C.1.A Planned Assessments

C.1.A.1 Field Sampling Technical System Audit

The USEPA and MPCA are responsible for the external technical system audits of field activities, including field sampling and measurements, for compliance of requirements specified for this project.

The Quality Assurance Manager and/or Field Team Leader of ENSR will be responsible for internal technical system audits to verify that field sampling procedures and field sampling measurements are properly followed. The technical system audits will include examination of

- Field sampling records,
- Field measurement results,
- Field instrument operating and calibration records,
- Sample collection, handling, and packaging procedures,
- QA procedures,
- Chain-of-custody,
- Sample documentation, etc.

Results of internal field technical system audits will be documented in quality control reports to management.

C.1.A.2 Fixed Laboratory Technical System Audits

The laboratory will perform internal systems audits as described in the laboratory quality assurance manual.

The City and/or Northwest Regional Quality Assurance Manager of ENSR (office in Fort Collins, Colorado), will be responsible for a fixed laboratory technical systems audit scheduled for 2002. Audit procedures will include both system audits and performance audits as necessary to satisfy the City that STL is capable of rendering satisfactory laboratory services under this QAPP. The audit will cover laboratory quality systems and analytical chemistry. The audit will be performed following the "City of St. Louis Park Audit Checklist" and will cover the following areas:

- QA organization and procedures, including internal audit results
- Personnel training and qualifications,
- Sample log-in procedures,
- Sample storage facilities,
- Analyst technique
- Adherence to laboratory SOPs and project QAPP,
- Compliance with QA/QC objectives,
- Instrument calibration and maintenance,
- Data recording, reduction, review, and reporting, and
- Cleanliness and housekeeping.

Preliminary results of the systems audit will be discussed with the Laboratory Director, Laboratory Operations Manager, Laboratory Project Manager, and Laboratory QA Manager. A written report that summarizes audit findings and recommends corrective actions will be prepared and submitted to the Laboratory QA Manager for response. The results of the audit, including resolution of any deficiencies, will be included in the QA reports to management, as described in Section C.2.

C.1.A.3 Performance Evaluation Sample Assessment

Performance evaluation samples are analyzed to verify the ability of the laboratory to correctly identify and quantitate compounds in check samples. These samples may be supplied internally or externally as blind or double-blind samples. These samples demonstrate data quality through statistical analysis. The results of internal performance audits may be used to document the training level of the analyst performing the work or to assess the overall performance of the facility. Periodic double-blind performance audits are conducted by STL to assess all aspects of laboratory performance from project initiation through analysis and reporting. These check samples will be reviewed as part of the biennial laboratory technical systems audit.

C.1.A.4 Data Validation Technical System Audits

Data validation and verification will be performed as described in Section D.2. In summary, a subset of data received will be subjected to a full data. The remainder of the data will receive a limited data validation. Data will be qualified and the results of the validation will be summarized in a validation memo. Each data validation technical systems audit will be reviewed by a validator other than the one performing the validation. This review will verify that the analytical deliverable package was complete and that any missing information requested from the laboratory was supplied, that validation worksheets were filled out accurately and completely, that validation actions were consistent with the validation guidelines established for this program and/or best professional judgement, and that the validation reports and data qualifiers accurately reflect the validation actions as documented on the worksheets.

C.1.A.5 Data Package Technical System Audits

Audits of analytical data packages will be conducted for 100% of the packages received as part of the data validation process (Section D.1). The review will include an evaluation of the package to ensure that (1) all required deliverables are provided, (2) the package contains the information necessary to reproduce the reported results, and (3) the QC acceptance criteria specified in the QAPP were met. Any deficiencies will be communicated to the laboratory and documented in the data validation reports. The laboratory QA Manager is responsible for performing data audits as specified in QA Policy No. QA-005.

C.1.A.6 Management System Review

The laboratory management system review is accomplished during the laboratory audit. ENSR conducts an internal, quarterly review of its projects for conformance with contractual and company policy requirements. ENSR's quarterly review includes an analysis of staffing and overall resources to meet project objectives. The review is conducted by ENSR's General Manager, who is the supervisor of ENSR's Project Manager. The ability of ENSR's project manager to meet all technical project requirements is also evaluated. The results of the quarterly review are maintained in ENSR's internal project files.

CITY OF ST. LOUIS PARK AUDIT CHECKLIST

Sample Receiving

YES NO

Are refrigerator/cold storage area temperatures recorded daily and are records properly maintained?
Comments:

Are sample chain-of-custody forms completed properly?
Comments:

Are the temperatures of the coolers being checked and recorded?
Comments

Are volatile samples stored separately?
Comments:

Is access to sample storage area restricted?
Comments:

Data Review

Are all calculations checked by the analyst for accuracy and completeness?
Comments:

Are anomalies documented and reported?
Comments:

What corrective actions are taken when the analytical results fail to meet QC criteria?
Comments:

Standard preparation

Are Class S weights used to check the balances?
Comments:

CITY OF ST. LOUIS PARK AUDIT CHECKLIST (cont.)

	YES	NO
Are non-EPA and non-NBS neat materials compared to EPA or NBS whenever possible? Comments:		
Have expired standards and reagents been discarded? Comments:		

Inorganics

Is the conductivity of the Milli-Q water system checked daily and recorded? Comments:		
Is linearity verified (correlation coefficient of at least 0.995) before sample analysis? Comments:		
If the CCV does not meet acceptance criteria, is the system recalibrated and are all affected samples reanalyzed? Comments:		

Organic Extraction

Are all reagents and solvents screened for potential contamination? Comments:		
What is the source of reagent water? Comments:		
Are spiking solutions and standards prepared from separate stocks? Comments:		
Is glassware cleaned appropriately? Comments:		

CITY OF ST. LOUIS PARK AUDIT CHECKLIST (cont.)

YES NO

Are the hood airflows checked and how often are they checked?
Comments:

GC/MS Lab

Are current SOPs available for all personnel in the area?
Comments:

Is preventive maintenance performed on all instruments?
Comments:

Have MDL studies been performed on all methods?
Comments:

Are method blanks analyzed with every batch of samples?
Comments:

Are results of QC samples verified to determine if QC criteria has
been met before sample analysis begins?
Comments:

Are QC results which are outside of acceptance limits checked for error?
Comments:

Are corrective actions taken as necessary and documented and samples
reprepped/reanalyzed?
Comments:

Are logbooks reviewed periodically, as indicated by the signature/date/comments
of the reviewer?
Comments:

C.1.B Assessment Findings and Corrective Action Responses

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out-of-limit QC performance that can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. All corrective action proposed and implemented should be documented in the QA reports to management (Section C.2). Corrective action should only be implemented after approval by the contractor Project Manager, or his designee.

Field Corrective Action

Corrective action in the field may be needed when the sample frequency is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP, etc.), or when sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. The field team may identify the need for corrective action. The Field Team Leader will approve the corrective action and notify the Project Manager. The Project Manager will approve the corrective measure. The Field Team Leader will ensure that the field team implements the corrective measure.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The QA auditor will identify deficiencies and recommend corrective action to the Field Team Leader. The Field Team Leader and field team will perform implementation of corrective actions. Corrective action will be documented in QA reports to the project management team.

Corrective actions will be implemented and documented in the field record book. Documentation will include:

- A description of the circumstances that initiated the corrective action,
- The action taken in response,
- The final resolution, and
- Any necessary approvals.

No staff member will initiate corrective action without prior communication of findings through the proper channels.

Laboratory Corrective Action

Corrective action in the laboratory are specified in laboratory SOPs and may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, and potentially high concentration samples may be identified during sample log-in or analysis. Following consultation with laboratory analysts and supervisory personnel, it may be necessary for the Laboratory QA Manager to approve the implementation of corrective action. If the nonconformance causes project objectives not to be achieved, the ENSR Project QA Officer will be notified.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action files, and in the narrative data report generated by the laboratory. If the corrective action does not rectify the situation, the laboratory will contact the ENSR Project QA Officer, who will determine the action to be taken and inform the appropriate personnel.

Corrective Action During Data Validation and Data Assessment

The need for corrective action may be identified during either data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives. If the data validator or data assessor identifies a corrective action situation, the Project Manager will be responsible for informing the appropriate personnel.

C.1.B.1 Other Corrective Actions

These sections discuss corrective actions which will be taken in the event that a sample or sample extract is lost or destroyed during shipment, storage or analysis, or in data audits.

C.1.B.1.1 Samples

In order to minimize the possibility of sample destruction during shipment, six 1-liter bottles will be taken for all Low-Level (ppt) samples. For all samples, field blanks and matrix spikes and duplicates, subsequent extraction and analysis will be conducted on four intact 1-liter bottle. All field blank duplicates will be extracted and held. In the event that the field blank is lost during analysis or invalidated, the duplicate field blank will be analyzed and reported. Additional sample matrix will be required for matrix spike analyses.

If less than four liters of a sample remains after shipment and storage for analysis, the Program Manager will be notified and another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

C.1.B.1.2 Samples Extracts

If a sample extract is broken or lost during analysis, the Program Manager will be notified and will be responsible for determining the need for replacing the lost sample. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section the action taken.

C.1.B.1.3 Quality Control Samples

If a method blank, or matrix spike and its duplicate is lost or broken during analysis, a replacement QC sample will be sampled and analyzed. The analysis report will clearly note that a replacement QC sample was analyzed.

If a field blank is lost or broken during shipment, storage, or analysis, its duplicate will be analyzed. The analysis report for the sample batch associated with the field blank will clearly note the occurrence in the discussion section.

C.1.B.1.4 Data Inconsistencies

Data inconsistencies are potentially short-term problems that are addressed by ENSR and the City jointly. During the previous years of CD-RAP monitoring, when any municipal well has contained PAH above advisory levels then the specific course of action to resample that well, in accordance with the CD-RAP was followed. The data have been successfully used for previous 13 years to identify "breakthrough" at the carbon treatment plants, and to plan the replacement of the carbon in accordance with the CD-RAP. Other than the resampling prescribed by the CD-RAP, data inconsistencies are not the basis for any field corrective actions. The long-term nature of the groundwater containment remediation strategy allows any data inconsistencies to be put into the context of a large data set that defines water quality. The laboratory analytical method has evolved, and has been refined, over the years to avoid data inconsistencies that were apparent during the earlier years of this program. The City and ENSR will continue to evaluate the laboratory analytical procedures in an effort to understand any data inconsistencies and the potential need for corrective actions.

C.2 Reports to Management

QA reports will be prepared by the ENSR Project QA Officer and submitted quarterly to the ENSR Project Manager. QA reports will document any problems identified during the sampling and analysis programs and the corrective measures taken in response. The QA reports are summarized in Table C-2. The QA reports will include:

- All results of field and laboratory audits,
- Problems noted and actions taken during data validation and assessment, and
- Significant QA/QC problems, recommended corrective actions, and the outcome of corrective actions.

D. DATA VERIFICATION/VALIDATION AND USABILITY

This element details the QA activities that will be performed to ensure that the collected data are scientifically defensible, properly documented, of known quality, and meet project objectives. Two steps are completed to ensure that project data quality needs are met:

- Data Verification/Validation
- Data Usability Assessment

All data generated through field activities, or through the analytical program, will be reduced and validated prior to reporting. No data will be disseminated until it has been subjected to the procedures summarized below.

D.1.A Field Data

The field data verification includes verification of sampling design, sample collection procedures and sample handling. Field data will be reviewed daily by the Field Team Leader to ensure that the records are complete, accurate, and legible and to verify that the sampling procedures are in accordance with the protocols specified in the Work Plan.

D.1.B Internal Laboratory Review

Prior to the release of any data from the laboratory, the data will be reviewed and approved by laboratory personnel. The review will consist of a tiered approach that will include reviews by the person performing the work, by a qualified peer, and by supervisory and/or QA personnel.

D.1.C Validation of Analytical Data

Analytical data validation includes the verification and validation of analytical procedures, quality control, calibration, and data reduction. Validation of the laboratory deliverables will be performed by ENSR. Data from the Reilly site will receive either full or limited data validation. Full validation will be performed on one data package of PAH (ppt) analyses and one data package of PAH (ppb) analyses from the Second Quarter sampling round. This round is chosen for the full validation since the levels of target analytes present the greatest challenge to the laboratory based on the range of concentrations and likelihood of detects. All other data packages and analyses will receive a limited validation. The validation levels are described below and summarized in Table D-1.

Full validation at a minimum, the data will be reviewed for the following:

- Completeness of deliverable,
- Technical holding times, and sample preservation
- Laboratory and field blank contamination,
- Surrogate spike recoveries,
- Field duplicates,
- MS/MSD recoveries and relative percent differences (RPDs),
- LCS recoveries,
- Initial and continuing calibrations,
- Instrument tuning,
- Internal standard performance,
- Calculation verifications
- Conformance of electronic deliverable to hard copy results.

Limited validation will include the following:

- Completeness of deliverable,
- Technical holding times, and sample preservation
- Laboratory and field blank contamination,
- Surrogate spike recoveries,
- Field duplicates,
- MS/MSD recoveries and relative percent differences (RPDs),
- LCS recoveries,
- Conformance of electronic deliverable to hard copy results.

Validation will be performed following "Region 5, Standard Operating Procedure for Validation of CLP Organic Data", (April 1991, Revised February 1997). These guidelines will be modified as necessary for the methods used during analysis of Reilly samples. Validation will be performed using the QC summary forms presented in the data package. The discovery of significant anomalies or discrepancies during validation using the summary forms may result in an in-depth review of the raw data and the incorporation of additional review elements into the validation of all data.

D.2 Validation and Verification Methods

D.2.A Field Data Verification

Field records will be reviewed by the Field Team Leader to ensure that:

- Logbooks and standardized forms have been filled out completely and that the information recorded accurately reflects the activities that were performed.
- Records are legible and in accordance with good recordkeeping practices, i.e., entries are signed and dated, data are not obliterated, changes are initialed, dated, and explained.
- Sample collection, handling, preservation, storage, and shipping procedures were conducted in accordance with the protocols described in the Work Plan, and that any deviations were documented and approved by the appropriate personnel.

D.2.B Laboratory Data Verification

Prior to being released as final, laboratory data will proceed through a tiered review process. Data verification starts with the analyst who performs a 100 percent review of the data to ensure the work was done correctly the first time. The data reduction and initial verification process must ensure that:

- Sample preparation and analysis information is correct and complete,
- Analytical results are correct and complete,
- The appropriate SOPs have been followed and are identified in the project records,
- Proper documentation procedures have been followed, and
- All nonconformances have been documented.

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data will be performed by an experienced peer or supervisor. This check will be performed to ensure that initial review has been completed correctly and thoroughly and will include a review of:

- Adherence to the requested analytical method SOP,
- Correct interpretation of chromatograms, mass spectra, etc.,
- Correctness of numerical input when computer programs are used (checked randomly),
- Correct identification and quantitation of constituents with appropriate qualifiers,
- Numerical correctness of calculations and formulas (checked randomly)

- Acceptability of QC data,
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.),
- Documentation of dilution factors, standard concentrations, etc.,
- Sample holding time assessment.

A third-level review will be performed by the Laboratory Project Manager before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. A narrative to accompany the final report will be prepared by the Laboratory Project Manager.

D.2.C Validation of Analytical Deliverables

Validation will be performed as described in Section D.1.C of the QAPP using the "Region 5, Standard Operating Procedure for Validation of CLP Organic Data", (April 1991, Revised February 1997). These guidelines will be modified to reflect any differences in analytical methodology and to incorporate the project-specific acceptance criteria defined in Section A.7 of this QAPP. See Table D-1 for a summary of the data validation to be performed.

Upon completion of the validation, a report will be prepared. This report will summarize the samples reviewed, elements reviewed, any nonconformances with the established criteria, and validation actions (including application of data qualifiers). Data qualifiers will be consistent with the U.S EPA Region 5 guidelines as shown below:

- J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- UJ - The analyte was not detected above the sample reporting limit; and the reporting limit is approximate.
- U - The sample was analyzed for, but was not detected above the sample reporting limit.
- R - The sample result is rejected due to serious deficiencies. The presence or absence of the analyte cannot be verified.

D.2.D Verification during Data Management

Data provided on diskette used to facilitate data handling will be verified against the hard copy data report during data validation.

D.2.E Calculation Techniques

The quality assessment procedures described above require calculations of relative percent difference (duplicate analyses) and percent recovery (matrix and surrogate spikes). The techniques for performing these calculations are described below.

- Precision - is the degree to which the measurement is reproducible. Precision is assesed by duplicate measurements by calculating the Relative Percent Difference (RPD) between duplicate measurements.

- Completeness - is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct normal conditions.

  To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the database is sufficient.

  When possible, the percent completeness for each set of samples is calculated as follows:

$$Completeness = \frac{valid\ data\ obtained}{total\ data\ planned} x\ 100\%$$

- Comparability - expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (ppm, ppb, etc.).

D.3 Usability/Reconciliation with Data Quality Objectives

This element describes how the verified/validated project data will reconcile with the project data quality objectives, how data quality issues will be addressed and how limitations on the use of the data will be reported and handled. The formal Data Quality Assessment consists of the following steps:

- Review of DQOs and Sampling Design
- Conduct Preliminary Data Review

- Select Statistical Test
- Verify Assumptions
- Draw Conclusions from the Data

The purpose of this section is to indicate the methods by which it will be ensured that the data collected for this investigation falls in line with the DQOs as described in Sections A.7 of this QAPP. To meet these DQOs, a combination of statistical procedures and qualitative evaluations will be used to check the quality of the data. These procedures will be used by the laboratory, in generating the data, and by the Data Validator, in the evaluation of the data for ultimate use in accordance with the CD-RAP.

The goal of this project is to produce data that can be used to define further the nature and extent of contamination and to establish the risk to human health of contaminants on the site. As such, the data generated must meet the data user's needs as defined in the project DQOs in Sections A.7 of this QAPP. The primary objectives for assessing the usability of the data are to ensure (1) data are representative of site conditions that can be combined with prior data; (2) data meet the project reporting limit requirements; and (3) data are of the quality needed in order to characterize the site accurately and precisely.

Results for QC samples, including field and laboratory blanks, spikes, and duplicates will be evaluated using the equations described below to determine the validity and usability of the data. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, contamination during sampling, contamination in the laboratory, and sample preservation and storage anomalies (i e. sample holding time or analytical instrument problems).

Data will be qualified for precision, accuracy, and comparability by the Data Validator. The Data Validator will apply the standard data validation qualifiers to data to indicate the level of uncertainty in the associated result. In general, for the purposes of the CD-RAP, data that are left unqualified, data qualified "U" (non-detected), data qualified "J" (detected as an estimated result), and data qualified "UJ" (non-detected at an estimated detection reporting limit) are considered valid and usable for project objectives. Data that are qualified "R" (rejected), due to severe exceedances of QC requirements, will be considered invalid and unusable for making project decisions.

D.4 Precision Assessment

The RPD, as a measure of variability between the matrix spike and matrix spike duplicate or sample and matrix duplicate (laboratory duplicates), and field duplicates, will be calculated to compare to precision and representativeness DQOs. The RPD of duplicate measurements is calculated according to the following formula:

$$\text{RPD} = \frac{(\text{Result in Sample 1 - Result in Sample 2})}{\text{Average(Result in Sample 1 and Result in Sample 2)}} \times 100$$

where:
Sample 1 = Initial sample or spiked sample result
Sample 2 = Duplicate sample or duplicate spiked sample result

In the event of precision results that do not meet the measuremetn performance criteria established for this project the results will be inspected to determine if the reduced precision can be attributed to sampling techniques (field duplicates) or sample contamination (field and laboratory blanks). If precision has been determined to be affected by sampling or contamination the data users must decide how to use data near the project action limits that may be affected. Data of reduced precision is expected to be usable with appropriate acknowledgement of the uncertainty associated with results that are near action levels.

D.5 Accuracy Assessment

Accuracy, as a measure of bias, will be evaluated based on the %Rs of the matrix spike sample, matrix spike duplicate sample, surrogates, internal standards, laboratory control sample, and initial and continuing calibration check samples. These QC results will be compared to the project measurement perfomance criteria for accuracy.

The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the %R.

Percent recoveries for spiked samples and QC are determined using the following equation:

$$\%R = \frac{\text{(Result in Spiked Sample - Result in Original Unspiked Sample)}}{\text{Known Amount of Spike Added}} \times 100$$

Percent recoveries for LCS and SRM are determined using the following equation:

$$\%R = \frac{\text{Result for compound in LCS or SRM}}{\text{Verified amount of compound in LCS or SRM from vendor information}} \times 100$$

Additionally, field and laboratory blanks will be used to evaluate whether field or laboratory procedures represent a possible source of contamination in the samples. Unmonitored contamination can allow false positive results to be reported and treated as true sample components when, in fact, they are not. This type of error will adversely affect the accuracy of the reported results. Several types of blanks, including field blanks, method blanks, and instrument blanks, will be used in this project as described in Section B.5.B.

Specific DQOs for blanks have been defined for this program in Sections B.5.B. In general, the procedure for assessing blank samples for potential contamination is as follows.

- Tabulate blank compound results.
- Identify blank samples for which compounds are reported above the method detection limits.
- If no compounds are detected above the method detection limits in any blanks, the associated data are reported unqualified and no blank actions are taken.
- If compounds are detected above the method detection limits in the blanks, the associated sample compounds will be qualified during data validation. This qualification may result in the negation of results at raised reporting limits due to blank actions.

Thus potential false results will be reported with elevated reported limits. These elevated limits will be recognized in the data available for the end user. Bias that does not meet the limits of the MPCs will be indicated by the results of LCS, MS, calibration, and surrogate analyses. Bias indicated by these MPCs will need to be evaluated to determine the effect on the use of the data. High bias on nondetect results, results that are well below action levels, or well over action levels may have little effect on the use of the data. Low bias for results that are well below the action levels or well over the action levels may have little effect on the use of the data. For results near the action levels with a high or low bias or indeterminate bias, the data will need to be reviewed carefully to establish if the data is usable for the intended purposes. Sample reanalysis, analysis of archived material, and/or recollection of the sample may be appropriate depending on criticalness of the missing data, logistical constraints, cost, and schedule.

D.6 Completeness Assessment

Completeness is the ratio of the number of valid sample results to the total number of results planned for collection. The goal of this program is to generate valid, usable data. However, in environmental sampling and analysis, some data may be lost due to sampling location logistics, field or laboratory errors, or matrix effects that may cause the rejection of results for some compounds. The overall completeness of collection of valid data is 95%. The Data Validator will assess the completeness of the overall data generation against the project goal of producing 95% of the planned data as valid and usable results for the CD-RAP. Valid and usable results are defined as those that are not rejected during validation (e.g. due to severe holding time, surrogate, or spike recovery noncompliances) or during the overall assessment (e.g. improper sampling technique). Following completion of the sampling, analysis, and data validation, the percent completeness will be calculated and compared to the project DQO of $\geq$ 95% using the following equation.

$$\% \text{ Completeness} = \frac{\text{number of valid/usable results obtained}}{\text{number of valid/usable results planned}} \times 100$$

If this goal is not met, data gaps may exist that will require evaluation to determine the effect on the intended use of the data. Sample reanalysis, analysis of archived material, and/or recollection of the sample may be appropriate depending on criticalness of the missing data logistical constraints, cost, and schedule.

D.7 Sensitivity

Sensitivity is evaluated by verifying that laboratory practical quantitation limits (LPQLs) meet the Project Quantitation Limits. The failure to calibrate with a standard at the LPQL or the presence of excessive dilutions may result in elevated detection limits. The effect of these elevated limits will need to be reviewed in light of the historical data and data collected during each quarter to determine if adequate information is available to satisfy the DQOs.

D.8 Comparability

Data comparability for samples obtained for this program will be assessed by the comparison to historical data for the same location. The acceptance criteria for comparing data split samples is a percent difference (%D) of 50% %D is calculated as

%D = 100*|(Conc 1 - Conc 2)/(average(Conc 1:Conc 2))

In the event of a comparability failure an effort will be made to establish whether the observed difference is due to the sample homogeneity, the sampling technique, or differences in laboratory technique

D.9 Overall Assessment of Environmental Data

Data assessment will involve data evaluation and usability to determine if the data collected are of the appropriate quality, quantity, and representativeness to support the CD-RAP. This evaluation will be performed by the Project Manager in concert with other users of the data. The QC results associated with each analytical parameter for each matrix type will be compared to the objectives presented in this QAPP. Data generated in association with QC results meeting these objectives and/or the data validation criteria will be considered usable. Data that does not meet the objectives and/or the data validation criteria may still be usable. This assessment may require various statistical procedures to establish outliers, correlations between data sets, adequate sampling location coverage of the site, etc, in order to assess the effect of qualification or rejection of data.

The effect of the qualification of data or loss of data deemed unacceptable for use, for whatever reason, will be discussed and decisions made on corrective action for potential data gaps.

Flowchart Figure D-1 indicates the steps that will be followed by the Project Manager in concert with other data users in the overall evaluation of data.

TABLES

TABLE A-1

Table of Reporting Limits for Tested Parameters

CAS Number	Compound	Reporting Limit Ng/L (PPT)	Reporting Limit ug/L (PPB)
271-89-6	2,3-Benzofuran	5.4	10
496-11-7	2,3-Dihydroindene	5 0	10
95-13-6	1H-Indene	4.7	10
91-20-3	*Naphthalene	8.6	10
4565-32-6	Benzo(b)thiophene	5.2	10
91-22-5	Quinoline	9.0	10
120-72-9	1H-Indole	4.7	10
91-57-6	2-Methylnaphthalene	5.9	10
90-12-0	1-Methylnaphthalene	5.6	10
92-52-4	Biphenyl	5.6	10
208-96-8	*Acenaphthylene	4.8	10
83-32-9	*Acenaphthene	5.7	10
132-64-9	Dibenzofuran	5.7	10
86-73-7	*Fluorene	4.1	10
132-65-0	Dibenzothiophene	4.1	10
85-01-8	*Phenanthrene	4.7	10
120-12-7	*Anthracene	3.4	10
260-94-6	Acridine	6.2	10
86-74-8	Carbazole	3.8	10
206-44-0	*Fluoranthene	4.6	10
129-00-0	*Pyrene	4.2	10
56-55-3	*Benzo(a)anthracene	4.3	10
218-01-9	*Chrysene	5.6	10
205-99-2	*Benzo(b)fluoranthene	4.7	10
207-08-9	*Benzo(k)fluoranthene	3.9	10
192-97-2	Benzo(e)pyrene	4.3	10
50-32-8	*Benzo(a)pyrene	2.5	10
198-55-0	Perylene	3.3	10
193-39-5	*Indeno(1,2,3-cd)pyrene	5.4	10

TABLE A-2

List of Carcinogenic PAH Compounds

Carcinogenic PAH[1]
benzo(a)anthracene
benzo(b)fluoranthene
benzo(j)fluoranthene
benzo(ghi)perylene
benzo(a)pyrene[2]
chrysene
dibenz(a,h)anthracene[2]
indeno(1,2,3-c,d)pyrene
quinoline

1 The total maximum levels of carcinogenic PAH established in the CD-RAP are:

Advisory Level - 15 ng/l
Drinking Water Criterion - 28 ng/l

2 The total maximum levels of the sum of benzo(a)pyrene and debenz(a,h)anthracene are:

Advisory Level - 3 0 ng/l (or the lowest concentration that can be quantified, whichever is greater)
Drinking Water Criterion - 5.6 ng/l

Table A-3

Data Quality Objectives for Analytical Measurements in Aqueous Samples

Parameter	Blanks[1]	LCS %R	MS/MSD %R	MS/MSD RPD
PAHs (ppt)				
Naphthalene	<PQL	20-150	20-150	50
Florene	<PQL	20-132	20-132	50
Chrysene	<PQL	20-132	20-132	50
IH-Indene	<PQL	20-150	20-150	50
Quinoline	<PQL	20-150	20-150	50
Benzo(e)pyrene	<PQL	20-150	20-150	50
2-methylnaphthalene	<PQL	20-150	20-150	50
PAH 8270C				
Acenaphthene	<PQL	60-101	60-101	30
Acenaphthylene	<PQL	60-100	60-100	30
Anthracene	<PQL	63-103	63-103	30
Benzo(a)anthracene	<PQL	61-104	61-104	30
Benzo(b)fluoranthene	<PQL	59-105	59-105	30
Benzo(k)fluoranthene	<PQL	60-105	60-105	30
Benzo(g,h,I)perylene	<PQL	55-112	55-112	30
Benzo(a)pyrene	<PQL	58-100	58-100	30
Chrysene	<PQL	59-102	59-102	30
Fluoranthene	<PQL	62-104	62-104	30
Fluorene	<PQL	62-104	62-104	30
Indeno(1,2,3-cd)pyrene	<PQL	56-112	56-112	30
Naphthalene	<PQL	58-98	58-98	30
Phenanthrene	<PQL	63-103	63-103	30
Pyrene	<PQL	61-105	61-105	30

[1] Applies to both laboratory and field blanks

TABLE A-4

Document Control

Item	Issued By	Issued To	Archived By
Field Notebooks	Field Coordinator	Sampling Team	Field Coordinator
Field Equipment Calibration Forms	Field Coordinator	Sampling Team	Field Coordinator
Sample Logs	Field Coordinator	Sampling Team	Field Coordinator
Chain-of-Custody Forms	Lab Sample Custodian	Field Coordinator	Lab Sample Custodian
Sample Labels	Field Coordinator	Sampling Team	Lab Sample Custodian

Table A-5

Method Detection Limit Report

Quanterra Environmental Services, Denver
METHOD DETECTION LIMIT STUDY - (Aqueous)

DATE COMPLETED:	4/30/99	PROG/PROJECT:	Instrument "C"
METHOD NUMBER:	PAH SIM	PROJECT NUMBER:	N/A
METHOD DESCRIPTION:	PPT5 PAHs	ANALYST.	M. Edwards
PREP METHOD:	4 Liter	QUALITY ASSURANCE:	T.Schumann

ANALYTE	SPIKE CONC ng/L	REPLICATE MEASUREMENT 1	2	3	4	5	6	7	AVG ng/L	Recovery of Spike %	PREC. ng/L	MDL ng/L	Report. Limit ng/L	STATUS*	
1H-Indene	5	3.642	3.679	3.220	3.674	2.288	3.168	3.502	3.3	65.89%	0.5	1.6	4.7	p	
1H-Indole	5	3.236	2.788	2.722	2.916	1.740	2.167	2.601	2.6	52.48%	0.5	1.6	4.7	p	
1-Methylnaphthalene	5	3.712	3.581	3.432	3.637	2.078	2.824	3.637	3.3	65.06%	0.6	1.9	5.6	p	
2, 3-Benzofuran	5	3.878	3.648	3.399	3.534	2.166	3.051	3.670	3.3	66.41%	0.6	1.8	5.4	p	
2,3-Dihydroindene	5	3.390	3.270	2.988	3.276	1.884	2.888	3.244	3.0	59.49%	0.5	1.7	5.0	p	
2-Methylnaphthalene	5	4.100	3.823	3.663	3.830	2.321	3.068	3.873	3.5	70.51%	0.6	2.0	5.9	p	
3-Methylcholanthrene	5	3.060	2.358	2.418	3.208	3.456	2.949	2.614	2.9	57.29%	0.4	1.3	3.9	p	
7,12-Dimethylbenz(a)anthracene	5	3.967	3.256	3.488	3.699	3.793	4.140	3.620	3.7	74.18%	0.3	0.9	2.8	p	
Acenaphthene	5	3.461	3.268	3.284	3.420	1.824	2.543	3.325	3.0	60.33%	0.6	1.9	5.7	p	
Acenaphthylene	5	3.055	2.864	2.701	2.862	1.686	2.052	2.856	2.6	51.62%	0.5	1.6	4.8	p	
Acridine	5	1.917	1.439	0.966	0.764	0.344	0.294	0.128	0.8	16.71%	0.7	2.1	6.2	p	w
Anthracene	5	2.988	2.666	2.664	2.884	3.130	2.012	2.633	2.7	53.85%	0.4	1.1	3.4	p	
Benzo(a)Anthracene	5	5.635	4.272	4.386	4.686	4.828	4.836	5.144	4.8	96.62%	0.5	1.4	4.3	p	
Benzo(a)pyrene	5	3.838	3.028	3.162	3.337	3.443	3.579	3.478	3.4	68.18%	0.3	0.8	2.5	p	
Benzo(b)fluoranthene	5	5.806	4.330	4.748	4.770	4.899	5.351	5.294	5.0	99.99%	0.5	1.6	4.7	p	
Benzo(b)thiophene	5	3.363	3.403	3.132	3.383	1.948	2.702	3.381	3.0	60.88%	0.5	1.7	5.2	p	
Benzo(e)pyrene	5	5.918	4.686	4.834	5.004	5.383	5.586	5.652	5.3	105.83%	0.5	1.4	4.3	p	
Benzo(g,h,i)perylene	5	7.710	6.270	6.142	6.683	6.489	7.616	7.399	6.9	138.06%	0.7	2.1	6.2	p	
Benzo(k)fluoranthene	5	5.426	4.327	4.166	4.582	4.331	4.588	4.626	4.6	91.56%	0.4	1.3	3.9	p	
Biphenyl	5	3.602	3.334	3.192	3.408	1.972	2.563	3.425	3.1	61.39%	0.6	1.9	5.6	p	
Carbazole	5	3.851	3.046	3.130	3.108	3.486	2.687	2.924	3.2	63.23%	0.4	1.3	3.8	p	
Chrysene	5	6.270	4.946	4.818	5.193	5.717	5.987	6.156	5.6	111.62%	0.6	1.9	5.6	p	

Table A-5

Method Detection Limit Report

Quanterra Environmental Services, Denver
METHOD DETECTION LIMIT STUDY - (Aqueous)

DATE COMPLETED:	4/30/99	PROG/PROJECT:	Instrument "C"
METHOD NUMBER:	PAH SIM	PROJECT NUMBER:	N/A
METHOD DESCRIPTION:	PPT5 PAHs	ANALYST:	M. Edwards
PREP METHOD:	4 Liter	QUALITY ASSURANCE:	T.Schumann

ANALYTE	SPIKE CONC ng/L	REPLICATE MEASUREMENT 1	2	3	4	5	6	7	AVG ng/L	Recovery of Spike %	PREC. ng/L	MDL ng/L	Report. Limit ng/L	STATUS*	
Dibenzo(a,h)anthracene	5	7.165	5 636	5.391	6.020	6.294	6:504	6.792	6.3	125.15%	0.6	2 0	5 9	p	
Dibenzofuran	5	3.654	3.419	3.366	3:463	1.972	2.774	3.554	3.2	63.43%	0.6	1.9	5 7	p	
Dibenzothiophene	5	3.932	3.258	3.432	3.561	3.936	2.603	3.543	3.5	69.04%	0.4	1:4	4 1	p	
Fluoranthene	5	4.348	3.516	3.751	3.666	4.291	2 940	3 519	3.7	74.37%	0.5	1·5	4 6	p	
Fluorene	5	3.789	3.161	3.587	3:548	3.665	2.497	3.432	3.4	67.65%	0.4	1:4	4.1	p	
Indeno(1,2,3-cd)pyrene	5	6.955	5.347	5.277	5.676	5.986	6 014	6.265	5.9	118.63%	0.6	1.8	5 4	p	
Naphthalene	5	5 001	4 656	4.185	4'514	2.603	5 535	4 365	4.4	87.91%	0.9	2 9	8 6	p	
Perylene	5	4.280	3.268	3.378	3.396	3.376	3 637	3 415	3 5	70.71%	0.3	1.1	3 3	p	
Phenanthrene	5	4.408	3.877	4.014	3.995	4.567	2 989	4 067	4 0	79.73%	0 5	1 6	4 7	p	
Pyrene	5	4.319	3.478	3 573	3.568	4 035	2.885	3.603	3.6	72.75%	0.5	1.4	4 2	p	
Quinoline	5	3.592	2.866	2.679	2.674	0.857	1.566	1.516	2.2	44.71%	0 9	3 0	9.0	p	w

* The calculated MDL was more than10 times less than the spiking level and may not be accurate. The reported MDL has been increased to exactly 10 times less than the spiking level.

MDL Spreadsheet Validation

Plug the values listed below into the spreadsheet to check that calculations and formulas are working properly.

MDL < 0.10X spike level	5	4.30	4.40	4.30	4.50	4.60	4.30	4.50	4.41	88.29%	0.12	0.38	5	f	
MDL > spike level	5	4.10	5.10	2.20	6.50	7.50	4.40	4.30	4.87	97.43%	1.73	5.43	10	f	
MDL > reporting limit	6	4.10	5.10	2.20	6.50	7.50	4.40	4.30	4.87	81.19%	1.73	5 43	5	f	
% recovery < 50%	10	4.10	5.10	2.20	6 50	7.50	4.40	4 30	4.87	48.71%	1.73	5.43	10	p	w
% recovery > 150%	5	7.25	8.10	7.50	7 60	7.50	7.75	7.60	7.61	152.29%	0.26	0.83	5	p	w
MDL meets all the above	5	4.40	4 25	4.40	4.60	4 90	4.30	4.20	4 44	88.71%	0.24	0.76	5	p	

TABLE B-1

Summary of Sampling and Analytical Program

Sample Matrix	Field Parameter	Laboratory Parameters	Estimated Number of Samples	Field Blanks	Field Duplicates	Matrix Spike[1]	Matrix Spike Duplicate[1]	Matrix Total
GAC Treated Water	None	PAH (ppt)	4	4	4	4	4	20
		Acid Fraction compounds[2]	1	X	1	1	1	4
GAC Feed Water	None	PAH (ppt)	1	X	1	1	1	4
Groundwater	pH, temperature, Specific Conductance	PAH (ppt)	58	11	11	11	11	102
		PAH (ppb)	58	11	11	111	11	102

1 **Matrix spike sample/matrix spike duplicate sample shall consist of the same matrix being analyzed. Triple the normal volume when related matrix spike/matrix spike duplicate samples are to be retrieved.**

2 Analysis of sample for acid fraction compounds shall be in accordance with EPA Method 625

TABLE B-2

Sampling Plan GAC Treatment System Monitoring Schedule[1]

RAP Section	Sampling Points	Start of Monitoring	Sampling Frequency	Analyses[2]
4.3.1(C)	Treated water (TRTD)	Date of plan approval	Quarterly	PAH (ppt)[3]
4.3.3(D)	Feed water (FEED)	Date of plan approval	Annually	PAH (ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH (ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625

1 This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 2000. Sections 4 and 12 of the RAP outline the additional monitoring that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring.

2 Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one every 10 samples or fewer. Treated water will be duplicated at a rate of 100 percent. Feed water duplicate samples will be collected and analyzed at a frequency of one per 10 samples.

3 ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry

TABLE B-3

Sampling Plan Groundwater Monitoring Schedule[a]

Source of Water	CD-RAP References	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]
Mt Simon-Hinckley Aquifer	5.1	SLP11, SLP12, SLP13, SLP17	Date of plan approval	Annually	PAH (ppt)[d]
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH (ppt)
Ironton-Galesville Aquifer	6.1.4	W105	Date of plan approval	Every even numbered year[h]	PAH (ppt)
	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH (ppt)
Prairie du Chien-Jordan Aquifer	Table 1[e]	SLP6, W48, W119, W413	Date of plan approval	Quarterly	PAH (ppt)
	Table 1	SLP4, SLP10 or SLP15, W23, W29, W40, W70, W401, W402, W403, E2, E3, E7, E13, E15	Date of plan approval	Annually	PAH (ppt)
	Table 1	E4, SLP5, SLP8, W32	Date of plan approval	Semi-annually	Water level monitoring[f]
	Table 1	H6, MTKA6, SLP7 or SLP9, SLP14, SLP16, W405 or W406[g]	Date of plan approval	Every even numbered year	PAH (ppt)
St. Peter Aquifer	Table 2[e]	SLP3, W24, W33, W122, W133, W410, W411, W412	Date of plan approval	Semi-annually	PAH (ppt)
	Table 2	W409	Date of plan approval	Semi-annually	8270C PAH
	Table 2	P116, W129, W408	Date of plan approval	Semi-annually	Water level monitoring
Platteville Aquifer	9.2.3	W421	Date of plan approval	Quarterly	8270C PAH[j]
	Table 2	W20, W27, W101, W131, W143, W426, W428, W431, W433, W434, W437, W438	Date of plan approval	Semi-annually	8270C PAH
	Table 2	W1, W18, W19, W100, W120, W121, W124, W130, W424	Date of plan approval	Semi-annually	Water level monitoring

TABLE B-3

Sampling Plan Groundwater Monitoring Schedule[a]

Source of Water	CD-RAP References	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]
Drift Aquifer	9.1.3	W420	Date of plan approval	Quarterly	8270C PAH
	Table 2	P109, P112, P307, P308, P309, P310, P311, P312, W11, W117, W136, W422, W427, W439	Date of plan approval	Semi-annually	8270C PAH
	Table 2	P47, W2, W10, W15, W116, W128, W135	Date of plan approval	Semi-annually	Water level monitoring

A This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 2001. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one for every 10 samples or fewer, and one duplicate sample will be collected for every 10 samples.

B Sampling points are located on the maps shown in Figures A-2 through A-6. Letter prefixes to well codes are defined as follows:

W	4-inch Monitoring Well
P	Monitoring Piezometer
SLP	St. Louis Park supply well
E	Edina supply well
H	Hopkins supply well
MTK	Minnetonka supply well

C Lists of parameters and descriptions of the methods for analysis of PAH and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analyses, except for those wells which prove to be inaccessible for such measurements.

D ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

E Wells will be sampled in accordance with Table 2 from the Agencies' October 3, 2000, letter, or Table 1 from the Agencies' October 19, 2000, letter, in lieu of the requirements given in the Consent Decree. Wells W19 and W130 were listed in the Agencies' Table 2 for both the Drift and Platteville Aquifers. Wells W19 and W130 are Platteville Aquifer wells.

F Water levels will be measured semi-annually at these wells in addition to the sampled wells, except for those wells that prove to be inaccessible for such measurements.

G W405 = American Hardware Mutual, W406 = Minikahda Golf Course.

H Wells sampled every two years shall alternate the season the well is sampled. For example, SLP14 will be sampled in fall 2002, spring 2004, fall 2006, etc.

I ppb = parts per billion. This signifies analysis by full scan 8270C.

J 8270C signifies USEPA SW846 Method 8270C for 16 PAH compounds.

TABLE B-4

Field Measurement Equipment Quality Control

Parameter	Device	Calibration	Routine Check		Control Limits
			Method	Frequency	
pH	Horiba U-10	Standardize in three or more standard buffer solutions, or use the auto calibration with factory solution	Calibration check-analyze standard buffer solution	1/10 samples	±0 1 pH units
			Analyze duplicates	1/10 samples	±0.1 pH units
			Auto calibration	1/10 samples	±0.1 pH units
Specific Conductance	Horiba U-10	Standardize using two or more KCL solutions or use the auto calibration with factory solution	Calibration check-analyze standard KCL solution	1/10 samples	±1 percent of range being used
			Analyze duplicates	1/10 samples	±1 percent of range being used
			Auto calibration	1/10 samples	±1 percent of range being used
Temperature	Horiba U-10	Factory calibrated	Not required	Not required	±0.1°C
Depth to Water	Water Level Measurement Device (Electric)	Factory calibrated	Not required	Not required	±0.01 Ft.

TABLE B-5

Sample Containers, Preservation Procedures, and Maximum Holding Times

Parameter	Containers[1]	Preservation[2]	Maximum Holding Time[3]
PAH (PPT)	Six 1-liter amber glass bottles, Teflon-lined caps	Cool to 4°C, protect from light	Seven days from sample collection until extraction, then 40 days after extraction
PAH (PPB and 8270C)	Two 1-liter amber glass bottles, Teflon-lined caps	Cool to 4°C, protect from light	Seven days from sample collection until extraction, then 40 days after extraction
Phenolics (Acid Fraction)	Two 1-liter amber glass bottle	Cool to 4°C	Seven days from sample collection until extraction, then 40 days after extraction

Ref: Federal Register Guidelines/Vol. 49, No. 209/Friday, October 26, 1984/ P. 43260

1 **Matrix spike samples shall consist of the same matrix being analyzed, therefore, triple the normal volume when a related matrix spike sample and matrix spike duplicate are to be retrieved.**

2 **Sample preservation will be performed immediately upon sample collection.**

3 **Samples will be analyzed as soon as possible after validated time of sample receipt (VTSR). The times listed are the maximum times that samples may be held before analysis and still be considered valid.**

Table B-6

Analytical Methodologies

Parameter	Methodology	SOP
PAH (parts per trillion)	SW-846 8270C SIM	DEN-MS-005 "PAHs by Selective Ion Monitoring for City of St. Louis Park"
PAH by 8270C	SW-846 8270C	DEN-MS-0001DEN "GC/MS Analysis Based on Method 8270C and 625"

TABLE B-7

Target Compounds and Key Ions for Low Level PAH Analyses

CAS No.	Compound	Quantitation Mass Ion	Confirmation Ion (Percent Abundance)
271-89-6	2,3-Benzofuran	118	90 (52)
496-11-7	2,3-Dihydroindene	117	118 (57)
95-13-6	1H-Indene	116	115 (108)
91-20-3	Naphthalene	128	102 (7)
4565-32-6	Benzo(B)Thiophene	134	89 (8)
91-22-5	Quinoline[1]	129	102 (20)
120-72-9	1H-Indole	117	90 (31)
91-57-6	2-Methylnaphthalene	141	115 (31)
90-12-0	1-Methylnaphthalene	141	115 (28)
92-52-4	Biphenyl	154	153 (35)
208-96-8	Acenaphthylene	152	151 (17)
83-32-9	Acenaphthene	154	153 (93)
132-64-9	Dibenzofuran	168	139 (40)
86-73-7	Fluorene	166	165 (90)
132-65-0	Dibenzothiophene	184	139 (19)
85-01-8	Phenanthrene	178	176 (19)
120-12-7	Anthracene	178	176 (19)
260-94-6	Acridine	179	178 (26)
86-74-8	Carbazole	167	166 (28)
206-44-0	Fluoranthene	202	200 (17)
129-00-0	Pyrene	202	200 (18)
56-55-3	Benzo(A)Anthracene[1]	228	226 (22)
218-01-9	Chrysene[1]	228	226 (26)
205-99-2	Benzo(B)Fluoranthene[1]	252	250 (22)
207-08-9	Benzo(K)Fluoranthene	252	250 (22)
192-97-2	Benzo(E)Pyrene	252	250 (35)

TABLE B-7

Target Compounds and Key Ions for Low Level PAH Analyses

CAS No.	Compound	Quantitation Mass Ion	Confirmation Ion (Percent Abundance)
50-32-8	Benzo(A)Pyrene[1]	252	250 (26)
198-55-0	Perylene	252	250 (24)
193-39-5	Indeno (1,2,3-CD)Pyrene[1]	276	274 (25)
53-70-3	Dibenz(A,H)Anthracene[1]	278	279(20)
191-24-2	Benzo(G,H,I)Perylene[1]	276	274 (25)
205-82-3	Benzo(J)Fluoranthene[1]	252	250 (22)

NOTE: The percent abundance for the confirmation ion is a typical value. Although these ratios will vary, the relative intensities of confirmation ions must agree within plus or minus 20 percent between the calibration standard for any given day and the samples run on that day.

1 Carcinogenic PAH as defined in Appendix A of the RAP

TABLE B-8

Target Compounds for Full Scan 8270C Analysis for PAHs

CAS No.	Compound	Reporting Limit ug/l
83-32-9	Acenaphthene	10.0
208-96-8	Acenaphthylene	10.0
120-12-7	Anthracene	10.0
56-55-3	Benzo(a)anthracene	10.0
205-99-2	Benzo(b)fluoranthene	10.0
207-08-9	Benzo(k)fluoranthene	10.0
191-24-2	Benzo(g,h,i)perylene	10.0
50-32-8	Benzo(a)pyrene	10 0
218-01-9	Chrysene	10.0
53-70-3	Dibenz(a,h)anthracene	10.0
206-44-0	Fluoranthene	10.0
86-73-7	Fluorene	10.0
193-39-5	Indeno(1,2,3-cd)pyrene	10.0
91-20-3	Naphthalene	10.0
85-01-8	Phenanthrene	10.0
129-00-0	Pyrene	10.0

TABLE B-9

Target Compounds for Extended Analyses

CAS No.	Compound	Reporting Limit
Other Carcinogenic PAH		**ng/l**
195-19-7	Benzo(c)phenanthrene	-
215-58-7	Dibenz(a,c)anthracene[1]	1.6
192-65-4	Dibenzo(a,e)pyrene	-
189-64-0	Dibenzo(a,h)pyrene	-
189-55-9	Dibenzo(a,i)pyrene	-
57-97-6	7,12-Dimethylbenz(a)anthracene	2.8
56-49-5	3-Methylcholanthrene	3.9
Acidic Compounds Listed in EPA Method 625		**µg/l**
108-95-2	Phenol	10
95-57-8	2-Chlorophenol	10
88-75-5	2-Nitrophenol	10
105-67-9	2,4-Dimethylphenol	10
120-83-2	2,4-Dichlorophenol	10
59-50-7	4-Chloro-3-methylphenol	10
88-06-2	2,4,6-Trichlorophenol	10
51-28-5	2,4-Dinitrophenol	50
100-02-7	4-Nitrophenol	50
534-52-1	4,6-Dinitro-2-methylphenol	50
87-86-5	Pentachlorophenol	50
1 Coelutes with dibenz(a,h)anthracene If these isomers are detected, they will be reported as a total value		

Table B-10

Internal QC Checks for Laboratory Analyses

Parameter	QC Check	Frequencies	Control Limits[1]	Laboratory Corrective Actions
PAH (ppt)	Method blanks	One per analytical batch, up to 20 samples	No target analytes above PQL	Reextraction/reanalysis of entire batch
	Surrogate spikes	Every sample, blank, standard prior to extraction	Per current laboratory control limits.	Reextract or flag data
	MS/MSD samples	One pair per analytical batch, up to 20 samples	Refer to Tables A-?.	Check LCS, reanalyze, flag results
	LCS	One per analytical batch, up to 20 samples	Refer to Tables A-?	Reextraction/reanalysis of entire batch
	Internal standards	Every sample, blank, standard prior to analysis	Area within 50-200% and RT within 0.5 min of IS in associated calibration standard	Reanalyze sample if no interference present
PAH 8270C	Method blanks	One per analytical batch, up to 20 samples	No target analytes above PQL	Reextraction/reanalysis of entire batch
	Surrogate spikes	Every sample, blank, standard prior to extraction	Per current laboratory control limits.	Reextract or flag data
	MS/MSD samples	One pair per analytical batch, up to 20 samples	Refer to Tables A-?.	Confirm with reanalysis, flag results
	LCS	One per analytical batch, up to 20 samples	Refer to Tables A-?	Reextraction/reanalysis of entire batch
	GC/MS tuning	At beginning of each 12 hour shift	Control criteria listed in SOP	Recalibrate instrument until control criteria are met
	Internal standards	Every sample, blank, standard prior to analysis	Area within 50-200% and RT within 0.5 min of IS in associated calibration standard	Reanalyze sample if no interference present

TABLE B-11

Instrument Maintenance Schedule Gas Chromatograph

Daily	As Needed
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed
Check inlets, septa. When using HP7673 autosampler, change septa daily.	Replace septum (approximately every 100 injections).
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Check reactor temperature of electrolytic conductivity detector.	Replace or repair flow controller in constant gas flow cannot be maintained.
	Replace fuse.
	Reactivate external carrier gas dryers.
	Detectors. clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace igniter. NPD: clean/replace collector assembly. PID: clean lamp window, replace seals. ECLD: check solvent flow weekly, change reaction tube, replace solvent, change reaction gas, clean/replace Teflon transfer line.
	Reactivate flow controller filter dryers when presence of moisture is suspected.
	HP7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.
	Purge and trap devices: periodic leak checks, replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), clean glassware.
	Purge and trap autosamplers: leak check system, clean sample lines, valves PTA-30 autosampler also requires cleaning the syringes, frits, valves, and probe needles, adjustment of micro switches, replacement of Teflon valve bleck, and lubrication of components.

TABLE B-12

Instrument Maintenance Schedule
Spectrophotometer

As Needed	Daily	Semi-Annually
Replace lamp	Re-zero the instrument	Perform wavelength calibration at 530 mm

TABLE B-13

Retention Times, Quantitation Ions and Internal Standards for Extended PAH List

Compound	Absolute Retention Time	Relative Retention Time	Quantitation Ions	Internal Standard
7,12-dimethylbenz(a)anthracene	18:46 minutes	0.97 minutes	M/Z 256	Perylene-d12
3-methylcholanthrene	19.46 minutes	1.023 minutes	M/Z 268	Perylene-d12

Table C-1

Planned Assessments

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) responsible for performing assessment, title and organizational affiliation	Person(s) responsible for responding to assessment findings, title and organizational affiliation	Person (s) responsible for identifying and implementing corrective actions (CA), title and organizational affiliation	Person (s) responsible for monitoring effectiveness of CA, title and organizational affiliation
Field Performance and System Audit[1]	Quarterly	Internal	ENSR	Bill Gregg/PM/ENSR	Bill Gregg/PM/ENSR	Bill Gregg/PM/ENSR	Bill Gregg/PM/ENSR
Laboratory Performance and System Audit[1]	Biennial	External	ENSR	Bill Gregg/PM/ENSR Rick Wellman/QA Chemist/ENSR	Larry Penfold/QA Manager/STL Denver	Devon Morgan/Operations Manager/STL Denver	Bill Gregg/PM/ENSR
Data Package Technical Systems Audit	Quarterly	External	ENSR	Bill Gregg/PM/ENSR Jim Herberich/Data Manager/ENSR	Brian Stringer/PM/STL-Denver	Brian Stringer/PM/STL-Denver	Bill Gregg/PM/ENSR
Data Validation Technical Systems Audit	Quarterly	External	ENSR	Rick Wellman/QA Chemist/ENSR	Brian Stringer/PM/STL-Denver	Rick Wellman/ QA Chemist/ENSR Devon Morgan/Operations Manager/STL Denver	Rick Wellman/QA Chemist/ENSR
Performance Evaluation Assessment	Biennial	External	ENSR	Bill Gregg/PM/ENSR Rick Wellman/QA Chemist/ENSR	Larry Penfold/QA Manager/STL Denver	Brian Stringer/PM/STL-Denver	Bill Gregg/PM/ENSR
Overall Usability Assessment	Quarterly	Internal	ENSR	Bill Gregg/PM/ENSR Rick Wellman/QA Chemist/ENSR	Bill Gregg/PM/ENSR Brian Stringer/PM/STL-Denver	Bill Gregg/PM/ENSR Rick Wellman/QA Chemist/ENSR	Bill Gregg/PM/ENSR

Table C-2

QA Reports

Type of Report	Frequency	Projected Delivery Date(s)[1]	Person(s) Responsible for Report Preparation, Title and Organizational Affiliation	Report Recipients, Title and Organizational Affiliation
Laboratory Performance and System Audit Report	Biennial	4th Qtr 2002	Rick Wellman/QA Chemist/ENSR and Bill Gregg/PM/ENSR	Brian Stringer/PM/STL and Scott Anderson/Superintendent of Utilities/SLP
Field Performance and System Audit Report	Quarterly	1 month after sampling	Peter Moore/Field Team Coordinator/ENSR	Bill Gregg/PM/ENSR
Data Package Technical Systems Audit Report	Quarterly	1 month following receipt of package	Jim Herberich/Data Manager/ENSR	Bill Gregg/PM/ENSR

Table D-1

Data Validation Summary Table

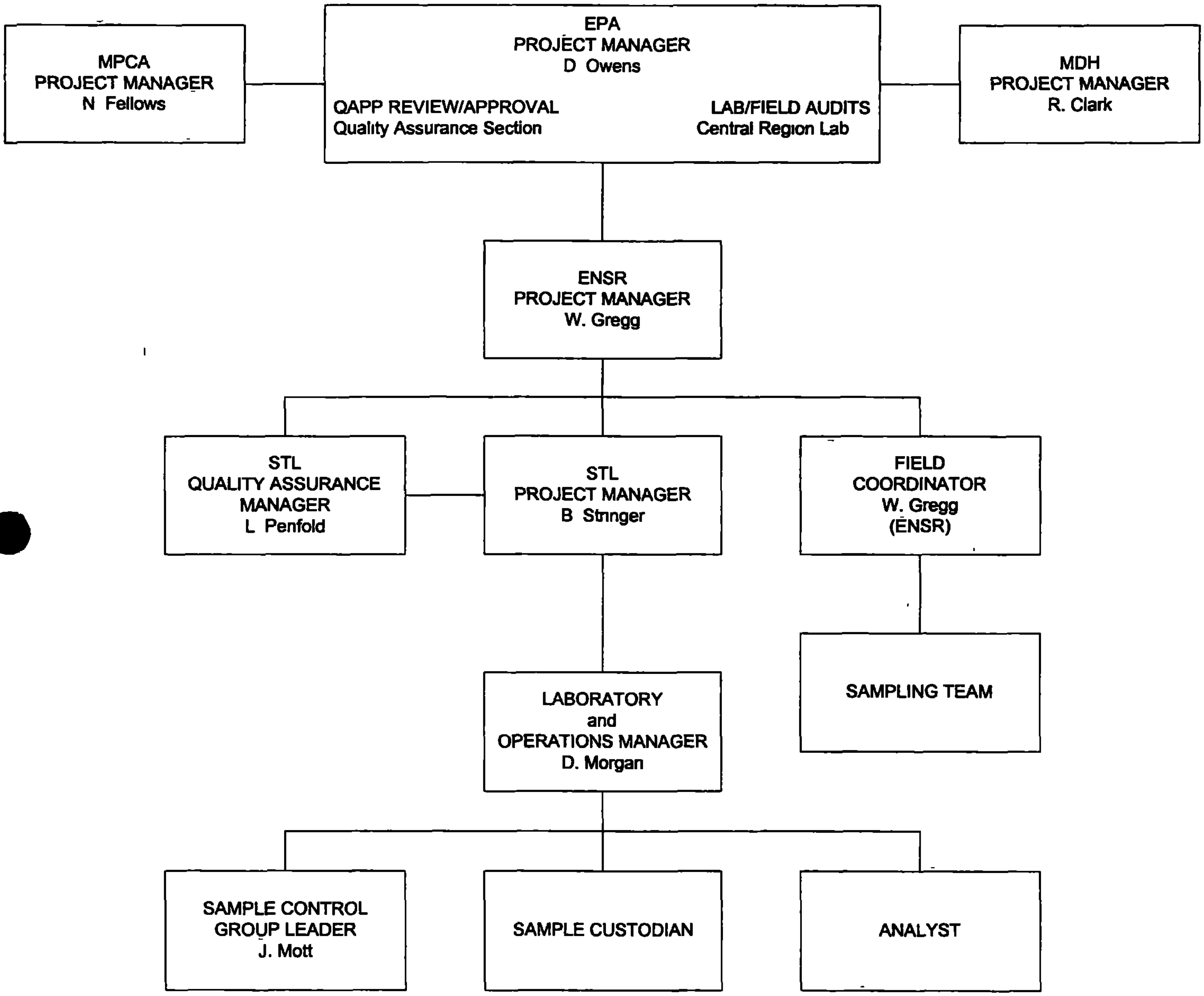
Medium/ Matrix	Analytical Parameter	Validation Criteria	Validation Criteria Modified for Method and QAPP	Data Validation Type	Data Validator (Name, title and organizational affiliation)	Responsibility for Data Validations (Name, title and organizational affiliation)
GAC Treated Water	PAH (ppt)	1	Yes	Limited	Data Validator, ENSR	Richard Wellman, Senior Project Chemist, ENSR
	Acid Fraction Compounds	1	Yes	Limited	Data Validator, ENSR	Richard Wellman, Senior Project Chemist, ENSR
GAC Feed Water	PAH (ppt)	1	Yes	Limited	Data Validator, ENSR	Richard Wellman, Senior Project Chemist, ENSR
Groundwater	PAH (ppt)	1	Yes	Limited/Full[2]	Data Validator, ENSR	Richard Wellman, Senior Project Chemist, ENSR
	PAH (ppb)	1	Yes	Limited/Full[2]	Data Validator, ENSR	Richard Wellman, Senior Project Chemist, ENSR

1 Region 5, Standard Operating Procedure for Validation of CLP Organic Data (April 1991, Revised Feb, 1997)

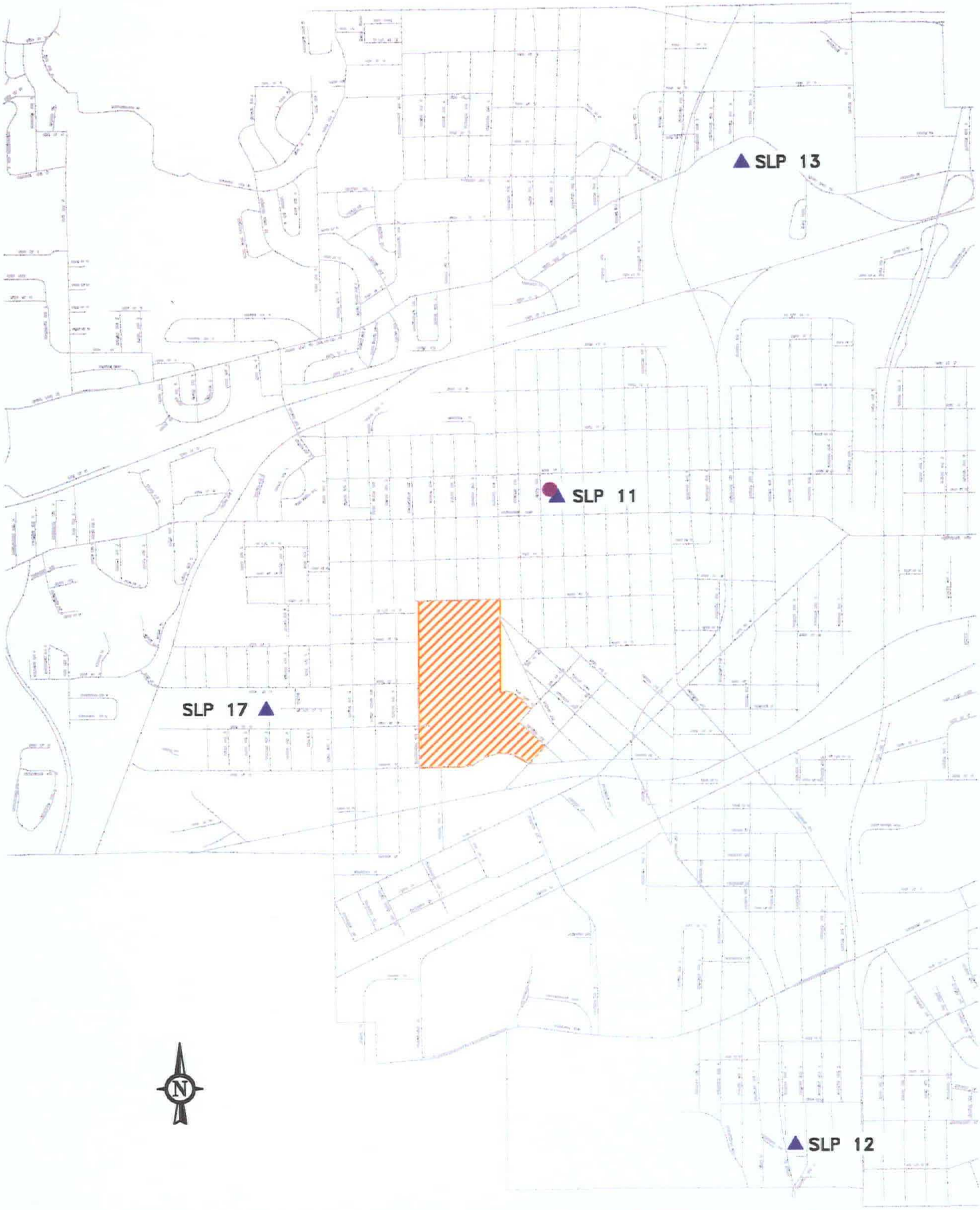
2 1 package from 2nd Quarter sampling to receive full validation

FIGURES

FIGURE A-1 Program Organization



J |Project\1620SLP\1620-013\Figures\orgchart vsd



REILLY SITE

LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

LOCATION OF GAC TREATMENT PLANT

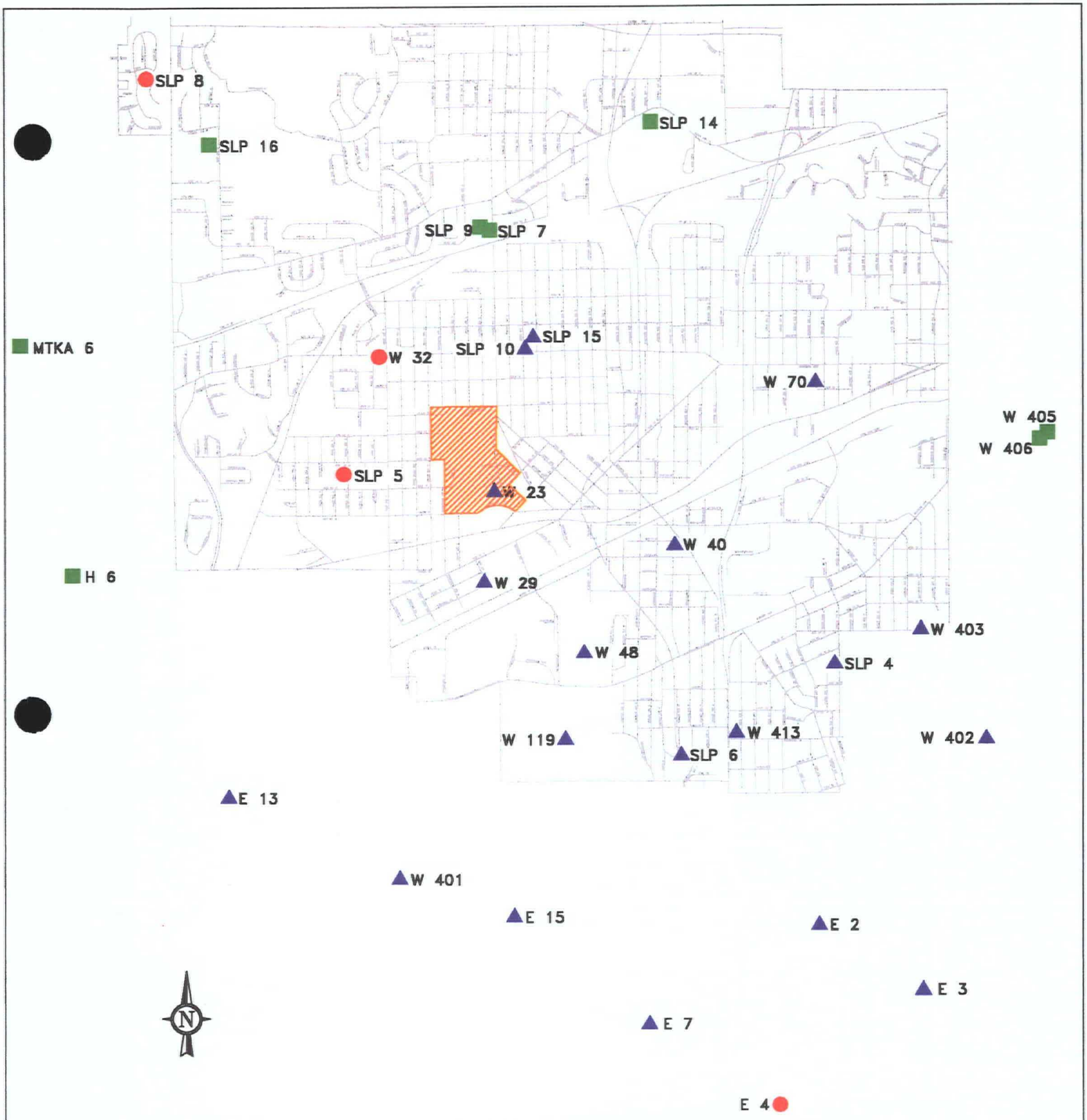
ENSR INTERNATIONAL

FIGURE A-2

LOCATION OF MOUNT SIMON HINKLEY AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN: C. BOEHM	DATE: 10/30/01	PROJECT NO.: 01620-013	REV:
FILE No.: FIG A-2 MSH.DWG	CHECKED: WMG		



REILLY SITE

LOCATION OF MONITORING WELL THAT IS SAMPLED ANNUALLY, SEMIANUALLY, OR QUARTERLY

LOCATION OF MONITORING WELL THAT IS SAMPLED IN EVEN NUMBER YEARS ONLY (I.E. WELL WILL BE SAMPLED IN 2002, 2004, ETC.)

LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

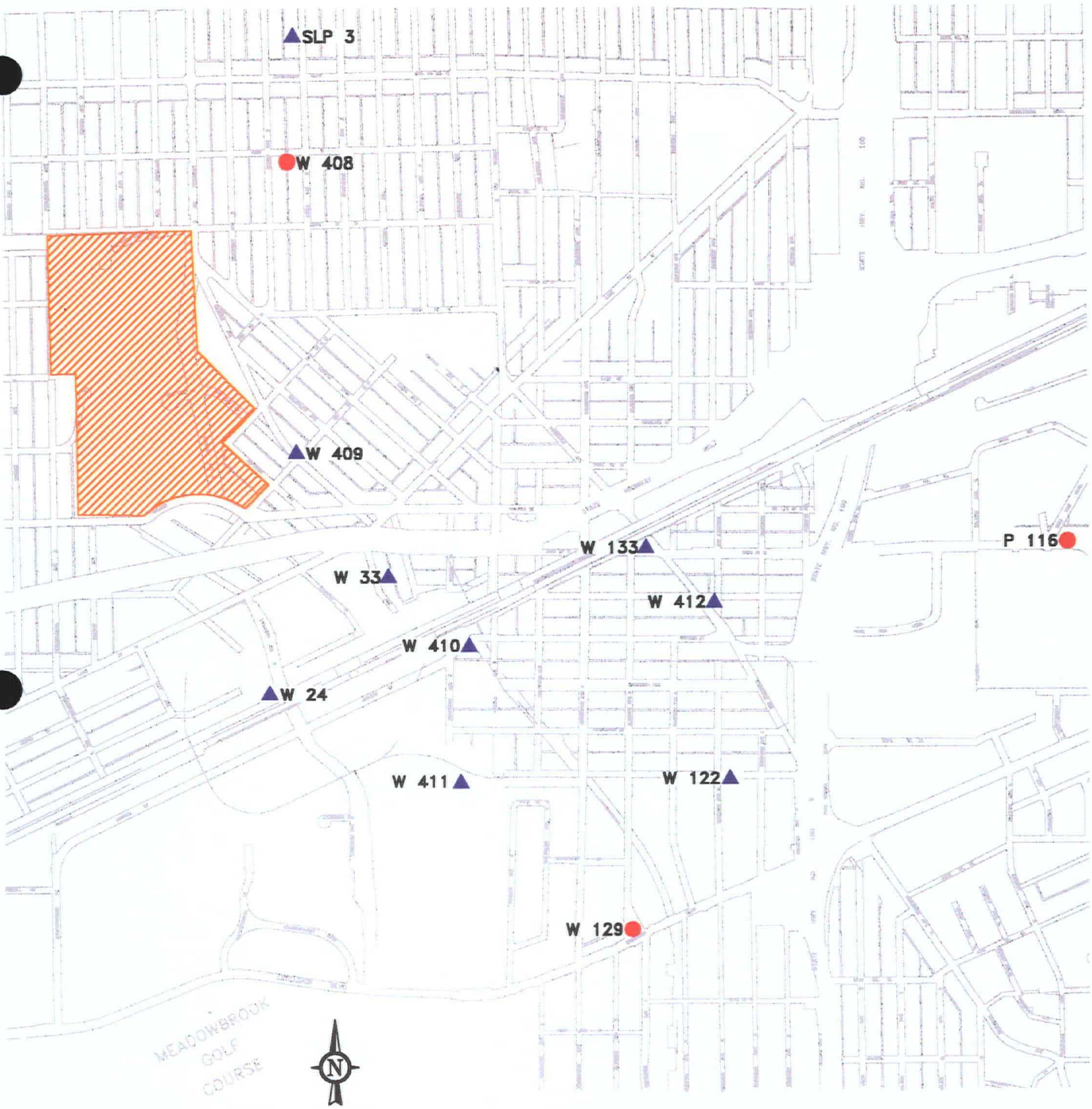
ENSR INTERNATIONAL

FIGURE A–3

LOCATION OF PRAIRIE DU CHEIN–JORDAN AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN: C. BOEHM	DATE: 10/30/01	PROJECT No.: 01620–013	REV:
FILE No.: FIG A–3 PCJ.DWG	CHECKED: WMG		



REILLY SITE

LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

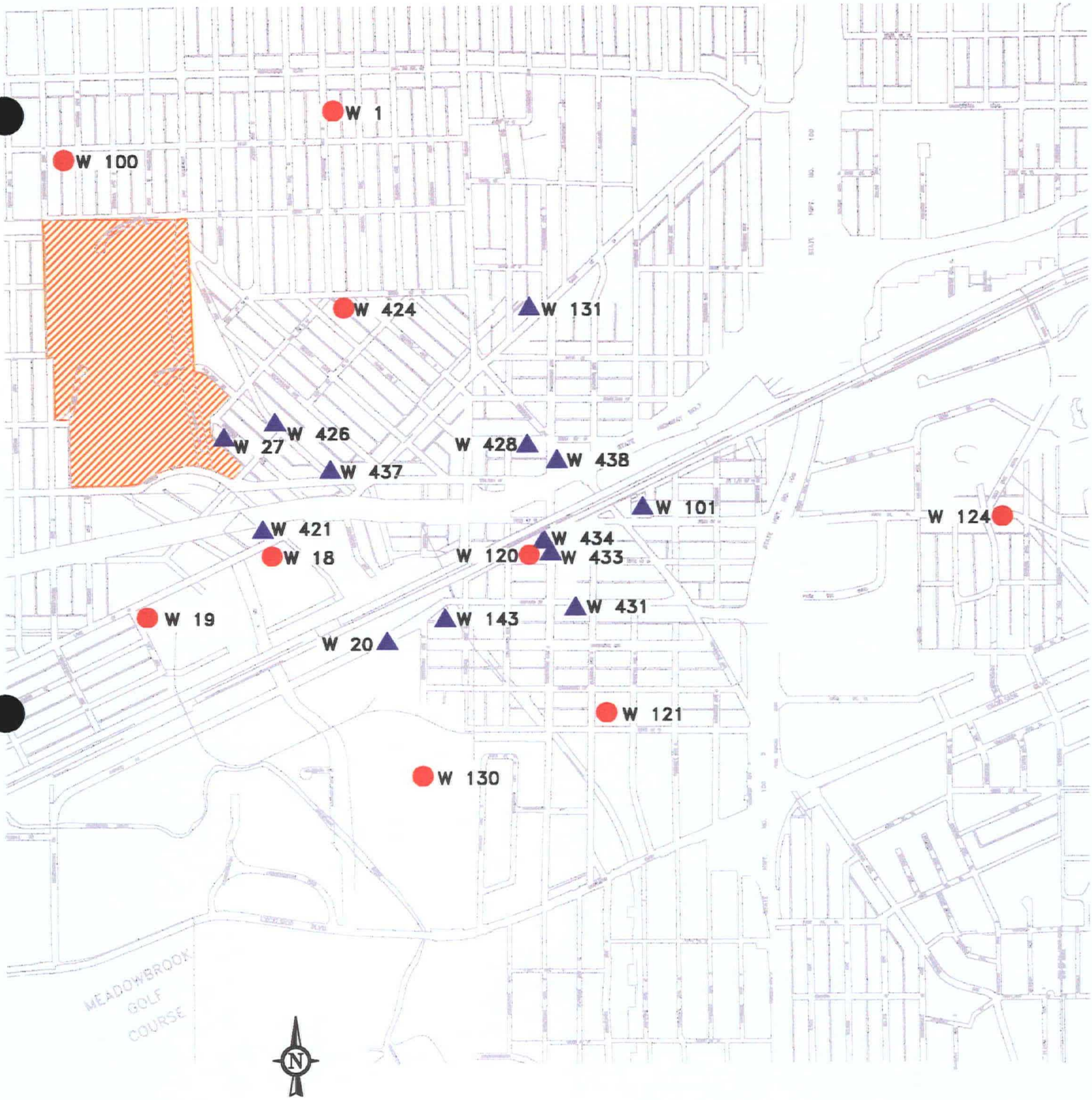
ENSR INTERNATIONAL

FIGURE A-4

LOCATION OF ST. PETER AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN:	C. BOEHM	DATE:	10/30/01	PROJECT No.:	01620-013	REV:
FILE No.:	FIG A-4 STP.DWG	CHECKED:	WMG			



REILLY SITE

LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

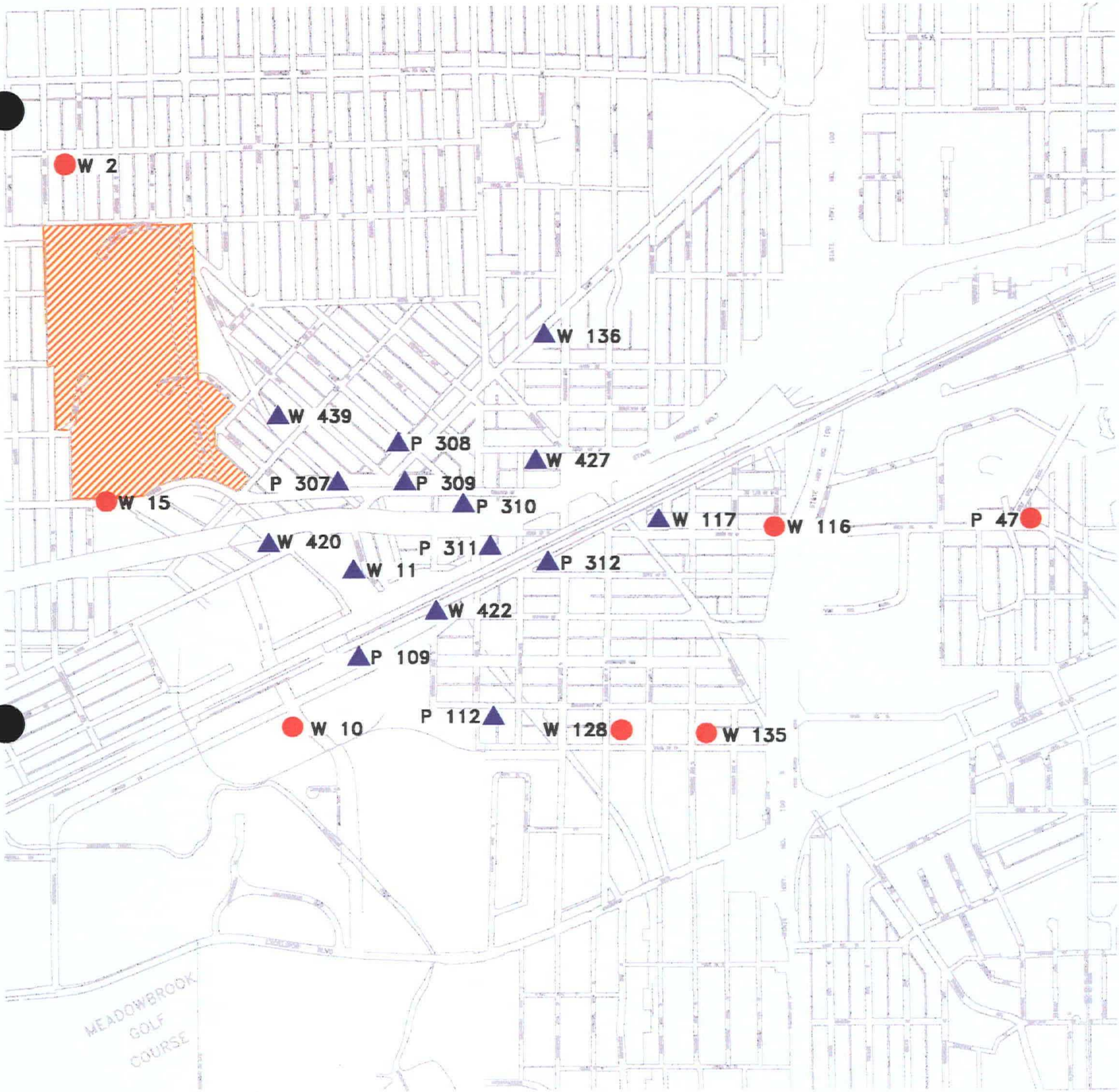
ENSR INTERNATIONAL

FIGURE A–5

LOCATION OF PLATTEVILLE AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN:	C. BOEHM	DATE:	10/30/01	PROJECT No.:	01620–013	REV:
FILE No.:	FIG A–5 PLV.DWG	CHECKED:	WMG			



REILLY SITE

LOCATION OF MONITORING WELL THAT WILL BE SAMPLED IN 2002

LOCATION OF MONITORING WELL THAT IS USED FOR GROUNDWATER LEVEL MONITORING ONLY

ENSR INTERNATIONAL

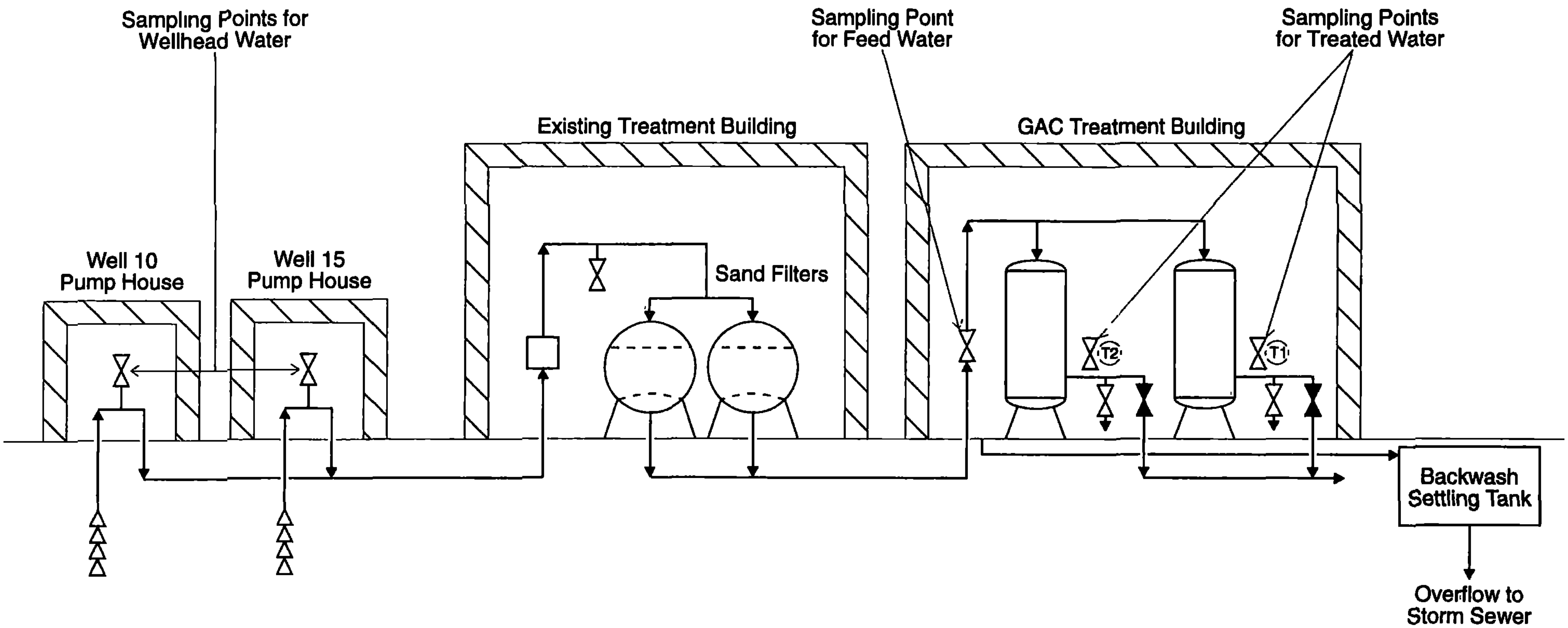
FIGURE A-6

LOCATION OF DRIFT AQUIFER GROUNDWATER MONITORING WELLS

2002 SAMPLING PLAN

DRAWN: C. BOEHM	DATE: 10/30/01	PROJECT No.: 01620-013	REV:
FILE No.: FIG A-6 DRI.DWG	CHECKED: WMG		

FIGURE B-1 GAC SAMPLING LOCATIONS



J \projects\1620 SLP\1620-013\figures\smploc cdr

B-2
Data Collection Process Flow Chart

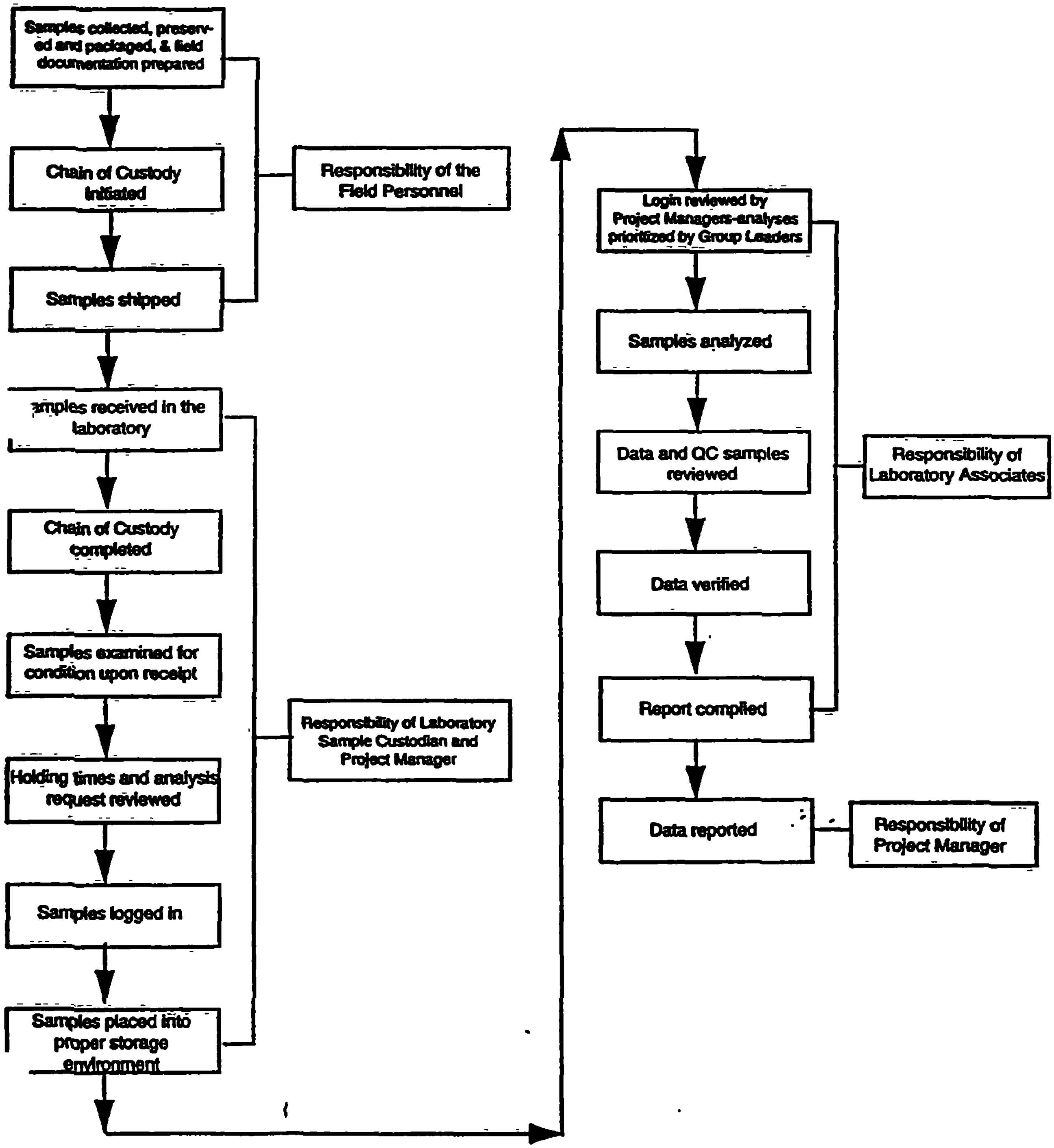
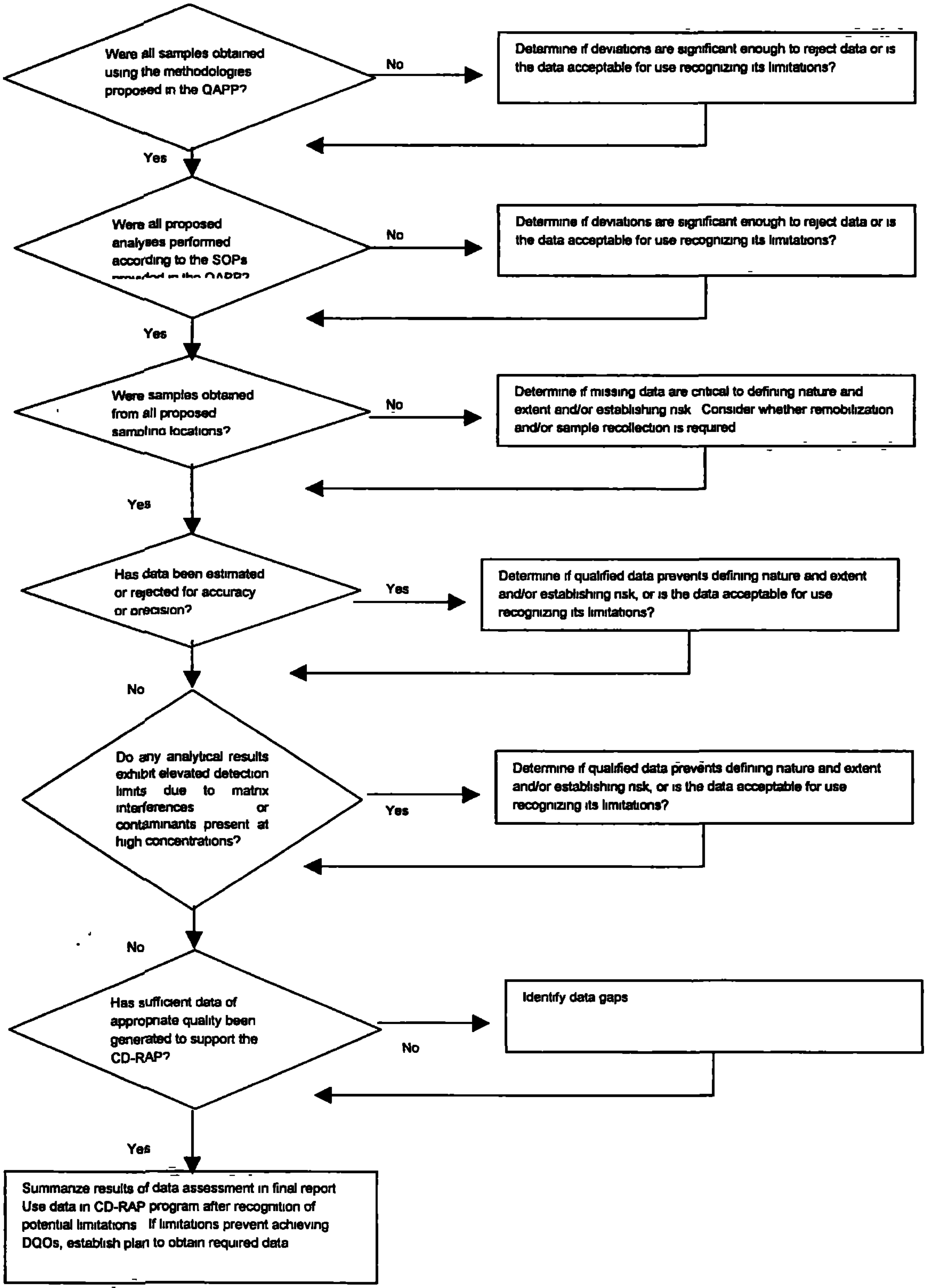


Figure D-1 Data Assessment Flow Chart



A

SOP NUMBER: 7121

ENSR

Field and Laboratory Measurement of pH

Date:	July 1998
Revision Number:	0
Author:	Lori Fuller
Discipline:	Water

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) provides basic instructions for routine calibration and operation of a variety of pH meters, including the Hydrolab, Hydac Multimeter Probe, Orion SA 230, YSI Model 3500, and Horiba U-10. Although these meters may measure additional parameters (e.g., temperature, specific conductivity, etc.), this SOP addresses pH measurement only (other capabilities are outlined in the appropriate SOP and manufacturer's individual instrument manuals). This SOP is designed specifically for the measurement of pH in accordance with EPA Method 150.1 and Standard Method 4500-H B which address electrometric pH measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan and/or Quality Assurance Project Plan (QAPP), hereafter referred to as the project plan, or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the pH meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- pH meter
- pH meter manufacturer's instruction manual
- Deionized water
- Clean glass or plastic beakers or cups
- 4.0, 7.0, and 10.0 buffer solutions
- Magnetic stirrer and Teflon-coated stirring bar
- Lint-free tissues
- Mild detergent and/or 10% hydrochloric acid (for use if samples contain oily material or particulate matter)
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

4.1.1 To achieve accurate pH measurements, samples should be analyzed in the field (preferably within 15 minutes), or as soon as possible after collection. Sample should be collected in plastic or glass containers.

4.1.2 After measuring a sample containing oily material or particulate matter, the electrode must be cleaned by carefully wiping with a lint-free cloth, or washing gently in a mild detergent, followed by a deionized water rinse. If this does not suffice, an additional rinse with 10% hydrochloric acid (followed by deionized water) may be needed.

4.1.3 As temperature can affect the pH measurements obtained, both the pH and the temperature of the sample must be recorded.

4.1.4 Calibration must include a minimum of two points that bracket the expected pH of the samples to be measured. An example of a calibration sheet is presented in Figure 1.

4.1.5 Primary standard buffer salts available from NIST can be purchased and are necessary for situations where extreme accuracy is required. Secondary standard buffers may be purchased as a solution from commercial vendors and are recommended for routine use. Buffers should not be used after their expiration dates as provided by the manufacturer. If the manufacturer does not supply an expiration date or if the buffers are prepared from pH powder pillows, etc., an expiration date of one year from purchase or preparation should be used. All standards must be labeled with manufacturer, lot number, and expiration date.

4.1.6 When using the meter in the laboratory, always place the buffer/sample beaker on the magnetic stirrer, and make sure the stirring bar is rotating during measurements. Rinse the stirring bar as well as the beaker between buffers/samples. CAUTION: The magnetic stirring plate can generate heat when used for an extended period of time, and lead to increased temperature of the buffers/samples.

EXCEPTION: Do not use the magnetic stirrer for acid rain samples. It is crucial not to induce dissolved gases into the sample to be absorbed or desorbed, as this will alter the pH. Stir the sample gently for a few seconds after introducing the electrode, then allow the electrode to equilibrate prior to recording temperature and pH readings.

4.1.7 When the meter is being used in the field, move the probe in a way that creates sufficient sample movement across the sensor; this insures homogeneity of the sample and suspension of solids. If sufficient movement has occurred, the readings will not drift (<0.1 pH units). Rinse the electrode with deionized water between samples and wipe gently with a lint-free tissue.

4.1.8 When measuring the pH of hot liquids, wait for the liquid to cool to 160°F or below.

4.1.9 Fluctuating readings may indicate more frequent instrument calibrations are necessary.

4.1.10 A "low sodium error" electrode may be used for samples with a pH greater than 10, to reduce sodium error.

4.2 Calibration and Measurement Procedures

4.2.1 The pH meter must be calibrated daily before any analyses are performed. The meter should be recalibrated every 12 hours or at the frequency specified in the project plan.

4.2.2 Connect the electrode to the meter. Choose either 7.0 and 10.0 (high range) or 4.0 and 7.0 (low range) buffers, whichever will bracket the expected sample range. Place the buffer in a clean beaker. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Measure and record the temperatures of the buffers using a calibrated thermometer or automatic temperature compensation (ATC).

4.2.3 Place the electrode into the 10.0 buffer or into the 7.0 buffer. (The calibration procedure described here begins with the buffer of the highest pH selected. The instrument manufacturer's calibration instructions may suggest starting with the buffer of the lowest pH selected).

4.2.4 Adjust the instrument calibration according to the manufacturer's instructions. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.

4.2.5 Refill the beaker with the 7.0 buffer or the 4.0 buffer. Rinse the electrode, gently wipe it with a lint-free tissue, and place it in the selected buffer solution. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Continue adjusting the instrument calibration according to the manufacturer's instructions. Record the electrode slope (if provided by the instrument) on the calibration sheet (an acceptable slope is between 92 and 102 percent). Measure and record the temperature of the buffer using a calibrated thermometer or ATC. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.

4.2.6 An additional check may be performed, if required by the project plan, by placing the electrode into an additional buffer solution. This buffer should be from a different source than the buffers used for the initial calibration. This buffer should read within +0.2 pH units of the buffer's true pH value.

4.2.7 Verify the calibration every 15 samples and after the last sample of the day with a buffer solution prepared from a different source than that used for initial calibration. Recalibrate the instrument if the check value varies more than 0.2 pH units from the true value.

4.2.8 The electrode will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analysis.

4.2.9 Recalibrate the instrument if the buffers do not bracket the pH of the samples.

4.2.10 The meter must be recalibrated following any maintenance activities and prior to the next use.

4.3 Troubleshooting Information

If there are any performance problems with the pH meter which result in inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

4.4.1 Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.

4.4.2 The electrode must be stored and maintained according to the manufacturer's instructions.

4.4.3 If an instrument with ATC is being used, the device should be checked on a quarterly basis for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

5.1 Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within +0.1 pH units.

5.2 The temperature readout of the meter will be checked annually against an NIST-traceable thermometer. If the difference is greater than 0.2°C, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.

5.3 Some regulatory agencies may require the analysis of USEPA Water Supply (WS) or Water Pollution (WP) performance evaluation (PE) samples. These PE samples will be analyzed as required.

6.0 DOCUMENTATION

6.1 All pH meter calibration, temperature check, and maintenance information will be recorded on a daily calibration sheet (Figure 1) or equivalent. pH data may be recorded on the appropriate laboratory or field data sheets or logbooks.

6.2 Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all buffer solutions
- Reading for pH 7.0 buffer before and after meter adjustment
- Reading for pH 4.0 or 10.0 buffer before and after meter adjustment
- Readings for all continuing calibration checks
- Temperature of buffers (corrected for any difference with reference thermometer), including units
- Slope reading (if provided by instrument)
- Comments

6.3 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and pH of sample (including units and duplicate measurements)
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform pH measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that pH measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Wastewater, 17th Edition. 1989.

USEPA. Methods for the Chemical Analysis of Water and Wastes (EPA 600/4-79-020). Revised 1983.

ENSR

Figure 1 Sample of Instrument Calibration Sheet

Water Quality Instrument Calibration										
Parameter	Instrument		Standard		Standard Value @25EC	Ambient Temp EC/EF	Initial Measured Value	Adjusted Measured Value	Initials Date/Time	Remarks
	Manuf/Model	Serial No	Manuf/Lot No	Exp Date						

All measured values must be corrected for temperature unless the instrument is operated in the ATC mode DO measurements must also be corrected for barometric pressure

Temp conversion: EC = 5/9(EF-32)

ENSR

SOP NUMBER: 7123

Field and Laboratory Measurement of Temperature	
Date:	July 1998
Revision Number:	0
Author:	Lori Fuller
Discipline:	Water

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) provides basic instructions for routine measurement of temperature using any high quality mercury-filled thermometer or thermistor with analog or digital read-out device such as the Hydrolab, Hydac Multimeter Probe, Seabird 911 CTD, and Horiba U-10. Multimeter instruments used for temperature measurement may measure additional parameters (e.g., conductivity, pH, etc.). This SOP addresses temperature measurement only (other capabilities are outlined in the appropriate SOP). This SOP is designed specifically for the measurement of temperature in accordance with EPA Method 170.1 and Standard Method 2550 B which address thermometric temperature measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan and/or Quality Assurance Project Plan (QAPP), hereafter referred to as the project plan, or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the temperature measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Thermometer or thermistor with analog or digital read-out device
- Manufacturer's instruction manual for the instrument
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Laboratory or field data sheets or logbooks
- Clear glass or plastic containers

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate temperature measurements, samples should be analyzed immediately upon collection (preferably within 15 minutes). Samples should be collected in glass or plastic containers.

4.2 Calibration and Measurement Procedures

4.2.1 ENSR-owned temperature measuring devices will, at a minimum, be checked annually as described in Section 5.0. The device will be checked against an NIST-traceable thermometer and the necessary compensation made for the difference in temperature between the two. Rental equipment will be checked by the manufacturer and documentation provided to ENSR. Certain oceanographic instruments such as the Seabird 911 CTD and the Coastal Microqual will be calibrated every 6 months in the manufacturer's laboratory using ITS90 standards. ITS90 is the International Temperature Scale developed by the Joint Panel of Oceanographic Tables and Standards. Post-cruise calibration records received from the manufacturer may be used to post-calibrate field data.

4.2.2 Immerse the thermometer or temperature measuring device into the sample.

4.2.3 Swirl and take a reading when the value stabilizes.

4.2.4 Record the temperature reading to the nearest 0.5° for a thermometer or 0.1° for digital meter-type instruments. For instruments other than the oceanographic instruments, compensate for any difference with the NIST-traceable thermometer.

4.2.5 Temperature measuring devices designed for *in situ* field measurement will be deployed in accordance with the manufacturer's instruction manual. For water-column profiling operations the sensor readings will be recorded manually in a designated field logbook or continuously through the use of a computer. An internal data-logger will be used for recording sensor measurements during moored deployment of a sensor. The frequency of data recording will be specified in the project plan. The location, date, and time of sensor deployment, along with depth (of measurement or mooring) will be recorded in conjunction with the temperature sensor data. Additional documentation requirements are listed in Section 6.2.

4.2.6 Temperature data may be post-calibrated using any of a variety of calibration data including, but not limited to, field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post calibration, and the technique used, will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with the meter-type temperature measuring device, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions. If performance problems exist with a thermometer, replace the thermometer.

4.4 Maintenance

Instrument maintenance for meter-type temperature measuring devices should be performed according to the procedures and frequencies required by the manufacturer.

5.0 QUALITY CONTROL

5.1 With the exception of the oceanographic instruments, the temperature measuring devices will, at a minimum, be checked against an NIST-traceable thermometer at the frequency stated in section 4.2.1. This verification procedure will be performed as follows:

- Immerse the thermometer or temperature sensor and the NIST-traceable thermometer into a sample.
- Allow the readings to stabilize.
- Record the readings and document the difference.
- Label the thermometer or temperature sensor with the correction value/adjustment and the date the accuracy check was performed.
- Compensate for the difference when sample measurements are taken.

5.2 Duplicate measurements of a single sample will be performed at the frequency stated in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within $\pm 0.5^{\circ}C$ or approximately $\pm 1.0^{\circ}F$.

6.0 DOCUMENTATION

6.1 Records for checking the accuracy of the thermometer or temperature measuring device (where applicable) will include:

- Date
- Thermometer or meter-type temperature measuring device checked
- Reference thermometer number
- Readings for reference thermometer and thermometer being checked
- Adjustment made for difference in readings
- Initials of analyst

6.2 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Thermometer ID number or instrument identification number/model
- Sample identification/station location
- Temperature of sample (including units and duplicate measurements) compensated for any difference with the reference thermometer if applicable
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform temperature measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that temperature measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Wastewater, 17th Edition. 1989.

USEPA. Methods for the Chemical Analysis of Water and Wastes (EPA 600/4-79-020). Revised 1983.

SOP NUMBER: 7124

Field and Laboratory Measurement of Specific Conductance

Date:	July 1998
Revision Number:	0
Author:	Lori Fuller
Discipline:	Water

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) provides basic instructions for routine calibration and operation of a variety of specific conductance meters, including the Hydrolab, Hydac Multimeter Probe, YSI Model 3500, Coastal Microqual, Horiba U-10 and Seabird 911 CTD. Although these meters may measure additional parameters (e.g., temperature, pH, etc.), this SOP addresses specific conductance measurement only (other capabilities are outlined in the appropriate SOP and manufacturer's individual instrument manuals). This SOP is designed specifically for the measurement of specific conductance in accordance with EPA Method 120.1 and Standard Method 2510 B which addresses specific conductance measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan and/or Quality Assurance Project Plan (QAPP), hereafter referred to as the project plan, or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the specific conductance meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Specific conductance meter
- Specific conductance meter manufacturer's instruction manual
- Deionized water
- Clean glass beakers or plastic cups
- Potassium chloride (KCl) solution, 0.01M, for determination of cell constant (0.5M KCl for saline water measurements)
- KCl standard at concentration that approximates sample concentrations
- Lint-free tissues
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets
- Laboratory or field data sheets or logbooks

4.0 4.0METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

4.1.1 Specific conductance measurements should be taken soon after sample collection since temperature changes, precipitation reactions, and absorption of carbon from the air can affect the specific conductance. If specific conductance measurements cannot be taken immediately (within 24 hours), samples should be filtered through a 0.45 μ filter, stored at 4°C and analyzed within 28 days.

4.1.2 Report results as specific conductance, μmhos/cm at 25°C.

4.1.3 As temperature can affect the specific conductance measurements obtained, record both the specific conductance and the temperature of the sample.

4.1.4 Secondary standards may be purchased as a solution from commercial vendors. These standards should not be used after their expiration dates as provided by the manufacturer. If the manufacturer does not supply an expiration date or if the standards are prepared from various salts (e.g., KCl), an expiration date of one year from purchase or preparation should be used. All standards must be labeled with manufacturer, lot number, and expiration date.

4.2 Calibration and Measurement Procedures

4.2.1 The specific conductance meter must be calibrated daily (or the calibration checked) before any analyses are performed. However, certain oceanographic instruments such as the Seabird 911 CTD and the Coastal Microqual, which, because of their sensitivity, are calibrated only by the manufacturer (at their specified frequency).

4.2.2 Set up the instrument according to the manufacturer's instructions.

4.2.3 Rinse the probe with deionized water and dry with a lint-free tissue.

4.2.4 Repeat the above procedure for the beakers or cups.

4.2.5 Pour a sufficient amount of the KCl standard (preferably at a concentration that approximates the sample concentrations) into the beaker or cup to cover the probe.

4.2.6 Immerse the probe in the standard.

4.2.7 Record the stabilized specific conductance reading of the standard and the temperature. Adjust the instrument reading (according to the manufacturer's instructions) to display the correct value of the standard. If the meter cannot be adjusted to display the correct value of the standard, the standard should read within 5% of the true value. If the meter reading is between 5% and 15% of the true value, calculate the cell constant using the formula below and correct all subsequent meter readings.

$$\textbf{Cell Constant} = \frac{\textbf{0.01M or 0.5M KCl Standard Conductance}}{\textbf{Conductance Meter Reading}}$$

If the meter reading exceeds the reference standard by greater than 15%, replace the instrument. If the meter does not have automatic temperature compensation (ATC), correct all measurements to 25°C by adding 2% of the reading per degree if the temperature is below 25°C and by subtracting 2% of the reading per degree if the temperature is above 25°C.

4.2.8 An additional check may be performed, if required by the project plan, by placing the probe into an additional KCl standard. This standard should be from a different source than the standard used for the initial calibration. This standard should read within 5% of the true value. Clean and rinse probe and cup as in Sections 4.2.3 and 4.2.4.

4.2.9 Verify the calibration every 15 samples and at the end of the day. Recalibrate or replace the instrument if the check value is not within 15% of the true value.

4.2.10 The probe will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analyses.

4.2.11 The meter must be recalibrated following any maintenance activities and prior to the next use.

4.2.12 Conductivity meters designed for *in situ* field measurement will be deployed in accordance with the manufacturer's instruction manual. For water-column profiling operations the sensor readings will be recorded manually in a designated field logbook or continuously through the use of a computer. An internal data-logger will be used for recording sensor measurements during moored deployment of a sensor. The frequency of data recording will be specified in the project plan. The location, date, and time of sensor deployment, along with depth (of measurement or mooring) will be recorded in conjunction with the DO sensor data. Additional documentation requirements are listed in Section 6.0.

4.2.13 Conductivity data may be post calibrated using any of a variety of calibration data including, but not limited to field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post-calibration, and the technique used will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with the specific conductance meter which result in inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

4.4.1 Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.

4.4.2 The probe must be stored and maintained according to the manufacturer's instructions.

4.4.3 If an instrument with ATC is being used, the meter should be checked annually for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

5.1 The meter must be calibrated daily before use and recalibrated every 12 hours, and will not be used for sample determinations of specific conductance unless the initial check standard value is within 5% of the true value.

5.2 Oceanographic instruments will be calibrated every 6 months in the manufacturer's laboratory using the functional relationship between salinity and conductance, temperature and pressure as defined by the Practical Salinity Scale of 1978 (PSS-78). Post-cruise calibration records received from the manufacturer will be used to post calibrate field data.

5.3 Duplicate measurements of a single sample may be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within 10%.

5.4 The temperature readout of the meter will be checked against an NIST-traceable thermometer at least quarterly. If the difference is greater than 0.2EC, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.

5.5 Some agencies may require the analysis of USEPA Water Pollution (WP) performance evaluation (PE) samples. These PE samples will be analyzed as required.

6.0 DOCUMENTATION

6.1 All specific conductance meter calibration, temperature check, and maintenance information will be recorded on the daily calibration sheet. (An example is presented as Figure 1). Specific conductivity data may be recorded on the appropriate laboratory or field data sheets or logbooks.

6.2 Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all standards
- Reading for standard before and after meter adjustment
- Readings for all continuing calibration checks
- Temperature of standards (corrected for any difference with reference thermometer)
- Cell constant value
- Comments

6.3 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and conductance of sample (including units and duplicate measurements) Note: show all calculations for converting instrument reading to µmhos/cm if the instrument provides readings in any other units. Useful conversions are: 1 mS/m = 10 µmho/cm or 1 µmho/cm = 0.1 mS/m.
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform specific conductance measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that specific conductance measurements be taken in the field by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Wastewater, 17th Edition. 1989.

USEPA. Methods for the Chemical Analysis of Water and Wastes, (EPA 600/4-79-020). Revised 1983.

Figure 1 Example of Instrument Calibration Sheet

Water Quality Instrument Calibration

Parameter	Instrument		Standard		Standard Value @25EC	Ambient Temp EC/EF	Initial Measured Value	Adjusted Measured Value	Initials Date/Time	Remarks
	Manuf/Model	Serial No	Manuf/Lot No	Exp. Date						

All measured values must be corrected for temperature unless the instrument is operated in the ATC mode DO measurements must also be corrected for barometric pressure.

Temp conversion: EC = 5/9(EF-32)

SOP NUMBER 7130

Groundwater Sample Collection from Monitoring Wells

Date:	November, 1999
Revision Number:	2
Author:	Charles Martin
Discipline:	Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This standard operating procedure (SOP) is concerned with the collection of valid and representative samples of groundwater from monitoring wells. The scope of this document is limited to field operations and protocols applicable during groundwater sample collection.

This SOP is written in a broad-based manner and considers the application of a variety of sampling equipment in the collection of representative groundwater samples. Respective state and/or federal agency regulations may require specific types of equipment to be used when applying this SOP to a particular project. The project manager should review the applicable regulatory requirements, if any, prior to the start of the field sampling program. Deviations from this SOP to accommodate regulatory requirements should be reviewed in advance of the field program and documented in the project work plan.

1.2 Quality Assurance Planning Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific QAPP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements typically suggest the collection of a sufficient quantity of quality control (QC) samples such as field duplicate, equipment and/or field blanks and matrix spike/matrix spike duplicate (MS/MSD) samples. These requirements should be outlined in the QAPP. Additional information regarding quality assurance sample collection relevant to groundwater sampling is contained in Section 5.0 of this SOP.

1.3 Health and Safety Considerations

Groundwater sampling may involve chemical hazards associated with the materials being sampled. Adequate health and safety measures must be taken to protect project sampling personnel from potential chemical exposures or other hazards.

These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

2.0 RESPONSIBILITIES

2.1 Project Manager

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project-specific work plan.

2.2 Sampling Technician

It is the responsibility of the sampling technician to be familiar with the sampling procedures outlined within this SOP and with specific sampling, quality assurance, and health and safety requirements outlined within project-specific work plans (Sampling Plan, HASP, QAPP). The sampling technician is responsible for collection of groundwater samples and for proper documentation of sampling activities as samples are being collected.

3.0 REQUIRED MATERIALS

Groundwater sampling objectives may vary significantly between projects. Project objectives should be defined within the project-specific work plans. The list of required materials below identifies the types of equipment which may be used for a range of groundwater sampling applications. From this list, a project-specific equipment list should be selected based upon project objectives and other factors such as the depth to groundwater, well construction, required purge volumes, and analytical parameters, among others. The various types of sampling equipment which may be used include:

Well Purging Equipment

- Bailers
- Bladder pumps
- Submersible pumps
- Peristaltic pumps
- Centrifugal Pumps
- Waterra™ pumps

Field Instruments

- Individual or multi-parameter meter(s) to measure temperature, pH, specific conductance, dissolved oxygen (DO) oxidation reduction potential (ORP), and/or turbidity
- Water level measuring device
- Interface probe or product detection paste

Sampling Equipment

- Reusable or disposable bailers
- Peristaltic pump
- Bladder pump

Sample Preparation Equipment

- Filtration equipment
- Intermediate containers
- Sample kit (i.e., bottles, labels, preservatives, custody records, cooler)

General Equipment

- Project-specific sampling plans (SAP, QAPP, HASP)
- Sample collection records
- Field notebook/pen
- Waterproof marker pens
- Deionized water dispenser bottler
- Sample cup
- Buckets
- Coolers, or sample shuttles
- Instrument calibration solutions
- Power source (generator of 12V marine battery)
- Equipment decontamination supplies (refer to SOP 7600)
- Health and safety supplies
- First-Aid kit
- Tool box

Expendable Materials

- Deionized water supply

- Disposable bailer string (nylon or polypropylene)
- 0.45 micron filters
- Paper towels
- Plastic sheeting
- Ice/blue ice for sample preservation
- Disposable latex powder-free glove liners
- Disposable nitrile gloves
- Plastic trash bags
- Ziplock® bags

This equipment list was developed to aid in field organization and should be used in preparation for each sampling event. Depending on the site-specific sampling plan, additional material and equipment may be necessary and should be determined before the scheduled sampling event. Similarly, not all of the items shown in this list may be necessary for any one sampling event.

Additional SOPs are also available which provide procedures for different aspects of groundwater sampling. These SOPs include:

- ENSR SOP 7121, Field and Laboratory Measurement of pH
- ENSR SOP 7122, Field and Laboratory Measurement of Dissolved Oxygen
- ENSR SOP 7123, Field and Laboratory Measurement of Temperature
- ENSR SOP 7124, Field and Laboratory Measurement of Specific Conductance
- ENSR SOP 7125, Field and Laboratory Measurement of Turbidity
- ENSR SOP 7131, Field Filtration of Water Samples for Inorganics
- ENSR SOP 7510, Packaging and Shipment of Samples
- ENSR SOP 7600, Decontamination of Equipment

4.0 METHOD

4.1 Instrument Calibration

Field instruments will be calibrated according to the requirements of the project-specific plan and water quality SOPs (see Section 3.0).

4.2 Sampling Preparation

Before opening the well, a clean working surface shall be set up around the well head using a plastic sheet with slit cut in the middle. Prior to opening the well, the required health and safety gear (as specified in the HASP) shall be donned. This, at a minimum, usually means wearing gloves to limit the potential for exposure to contaminants as well as reduce the potential for handling-induced contamination of sampling equipment.

4.3 Well Security and Condition

At each monitoring well location, observe the conditions of the well and surrounding area. The following information shall be noted on the Groundwater Sample Collection Record (Attachment 1 or 2) or in the field notebook:

- Condition of the wells identification marker
- Condition of the well lock and associated locking cap
- Integrity of the well - protective outer casing, obstructions or kinks in the well casing, presence of water in the annular space, and the top of the interior casing
- Condition of the general area surrounding the well

4.4 Measuring Point Determination

Before collecting a water level measurement, check for an existing measuring point (notch, or other visible mark) established either at the time of well installation or by the latest survey. Generally, the measuring point is referenced from the top of the well casing (TOC), not the protective casing. If no measuring point exists, a measuring point should be established, clearly marked, and identified on the Groundwater Sample Collection Record or the field logbook. The same measuring point should be used for subsequent sampling events.

4.5 Free Product Determination

Wells that may potentially contain free product should be assessed for product with an interface probe or product detection paste. Interface probes generally operate on the same principle as a water level tape although they are designed to register water and product levels usually with different audible tones. Product paste generally is used in combination with some type of measuring tape which is lowered into the well with a coating of paste applied to it. Wells containing free product are generally not used for groundwater sampling, since the concentration of contaminants present in the free product can adversely effect the quality of the water sample, lending to a non-representative water sample.

4.6 Water Level Measurement

To obtain a water level measurement, lower the probe of a water level measuring device into the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. At this time the precise measurement should be determined (to nearest 0.01 feet) by repeatedly raising and lowering the tape to converge on the exact measurement. Obtain the reading of the TOC measuring point. The water level measurement should be entered on the Groundwater Sample Collection Record or in the field records.

The measurement device shall be decontaminated immediately after use with a non-phosphatic detergent and rinsed with distilled water. Generally, only that portion of the tape which enters the water table should be cleaned. It is important that the measuring tape is never placed directly on the ground surface or allowed to become kinked. Measuring devices, including interface probes, which come into contact with free product will likely require more thorough decontamination (see SOP 7600).

4.7 Purge Volume Calculation

Wells designated for sampling require purging to remove stagnant water in the well. A single casing volume of groundwater will be calculated after measuring the length of the water column and checking the well casing diameter. The Groundwater Sample Collection Record provides information used to compute the casing volume, which includes: a diagram, a numerical conversion table, and the standard calculation. The volume of standing water in the well (ie., one purge volume) should be entered on the Groundwater Sample Collection Record.

4.8 Well Purging Methods and Procedures

4.8.1 Objectives

Prior to sample collection, purging must be performed for all groundwater monitoring wells to remove stagnant water from within the casing and gravel pack and to ensure that a representative groundwater sample is obtained.

There are three general types of non-dedicated equipment used for well purging and include: bailers, surface pumps and down-well pumps. The purge method and equipment selected should be specified in the project-specific work plans.

NOTE: This SOP only describes the most common equipment and methods used for purging. Other purging equipment, as well as dedicated equipment, can be used provided that the method employed does not have an adverse affect on the overall quality of the groundwater.

Regardless of the purge method, purge water temperature, pH, and specific conductance will be monitored at predetermined purge volumes and recorded on the Groundwater Sample Collection Record. Additional water quality parameters may be required by the project-specific sampling plan. In general, purging will be considered complete following the withdrawal of at least 3 to 5 well volumes of groundwater and when all field parameters have stabilized to within 10% of their preceding measurements.

Purging a well to dryness may occur under some low-yield conditions. When the well recovers, a cascading effect may occur within the screened zone which can volatilize some organic compounds. This may be considered inappropriate by regulatory agencies when volatile organic compounds (VOC) are the target analyte of interest. Purging a well to dryness, then sampling after it has recovered may be acceptable for other target analytes, however. Under low yield conditions, low-flow sampling pumps such as bladder pumps may be required for VOC sample collection.

4.8.2 Bailing

General

Bailing is often the most convenient method for well purging especially if only a small volume of purge water is required during the purge routine. Bailers are constructed using a variety of materials including PVC, polyethylene, stainless steel, and Teflon®. Teflon® bailers are generally most "inert" and are available in reusable and disposable form. Disposable polyethylene bailers are relatively inert and inexpensive. Reusable stainless steel and PVC bailers must be decontaminated between uses. Most commercially available bailers are constructed to fit into a 2-inch diameter well, although other bailer diameters are available.

Waterra™ foot valves are essentially bailer check valves which manually thread onto the bottom of standard pump tubing (polyethylene, teflon). The foot valves are commercially available in a variety of diameters in stainless steel, Teflon®, and high-density plastic (Delrin). The foot valves operate by manually or mechanically raising and lowering the valve assembly within the water column which raises the water level within the discharge tube. Flow rates usually in the vicinity of 1 gallon per minute can be achieved with these devices.

Measurements of the pumping rate, temperature, pH, and specific conductance (and/or other parameters as required) should be made after each purge volume is removed and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been withdrawn and the field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

Bailing presents two potential problems with well purging. First, increased suspended solids may be present in samples as a result of the turbulence caused by raising and lowering the bailer through the water column. High solids concentrations may affect sample representativeness. Second, bailing may be less feasible for deep wells or wells which require a large volume of water to be removed during purging because of the time involved with continuous insertion and removal/emptying of the bailer.

Bailing Procedure

Obtain a clean bailer and a spool of clean polypropylene or nylon bailer cord. Uncover the top end of the bailer and tie a bowline knot, or equivalent, through the bailer loop. Test the knot and the bailer itself to ensure that all knots and parts are secure prior to inserting the bailer into the well.

Remove the protective wrapping from the bailer, and lower the bailer to the bottom of the monitoring well and cut the cord at a proper length. Bailer rope should never touch the ground surface at any time during the purge routine. Tie a hand loop at the end of the bailer cord.

Raise the bailer by grasping a section of cord using each hand alternatively in a "rocking" action. This method requires that the sampler's hands be kept approximately 2-3 feet apart and that the bailer rope is alternately looped onto or off each hand as the bailer is raised and lowered.

Grab the bailer with one hand as it emerges from the well. Pour the bailed groundwater from the bailer into a graduated bucket to measure the purged water volume. Repeat this procedure until one complete purge volume of water is removed from the well.

At the end of one complete well purge volume, place a small of purged water into a sample cup. Measure temperature, pH and specific conductance (and for other assigned parameters) and record the results on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been withdrawn and the specific field parameters have stabilized to within 10% of their preceding measurement.

4.8.3 Surface Pumps

General

Well purging using pumps located at the ground surface can be performed with peristaltic or centrifugal pumps if the water level in the well is within approximately 20 feet of the top of the well.

Peristaltic pumps provide a low rate of flow typically in the range of 0.02-0.2 gallons/minute (75-750 ml/min). For this reason, peristaltic pumps are not particularly effective for well purging. Peristaltic pumps are suitable for purging situations where disturbance of the water column must be kept minimal for particularly sensitive analyses.

Centrifugal pumps are designed to provide a high rate of pumping, in the range of 5 to 40 gallons/minute (gpm), depending on pump capacity. Discharge rates can also be regulated somewhat, provided the pump has an adjustable throttle. These pumps also require polyethylene or teflon-lined polyethylene tubing as suction line. The pump may also require priming to initiate flow.

Peristaltic Pump Procedure

Attach a new suction and discharge line to the peristaltic pump. Silicon tubing must be used through the pump head and must meet the pump head specifications. A second type of tubing may be attached to the silicon tubing for use as the suction and discharge lines. The secondary tubing material, usually consisting of polyethylene or teflon-lined polyethylene, should be compatible with the target analytes. The suction line must be long enough to extend to the static groundwater surface and reach further should drawdown occur during pumping.

Measure the length of the suction line and lower it down the monitoring well until the end is in the upper foot or more of the water column. Start the pump and direct the discharge into a graduated bucket. Adjust the pumping rate with the speed control knob so that a smooth flowing discharge is attained.

Measure the pumping rate in gallons per minute by recording the time required to fill a calibrated bucket. The pumping shall be monitored to assure continuous discharge. If drawdown causes the discharge to stop, the suction line will be lowered very slowly further down into the well until pumping restarts.

Measurements of temperature, pH and specific conductance (and/or other assigned parameters) should be made after each well purge volume and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been removed and the specific field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

<u>Centrifugal Pump Procedure</u>

Attach a new suction and discharge line to the centrifugal pump. Start the pump and record the stabilized rate of discharge. As with other well purging systems, measurement of temperature, pH, and specific conductance (or other parameters as required) will be made after each well purge volume has been removed. These measurements shall be recorded on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been removed and the field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

4.8.4 Down-Well Pumps

<u>General</u>

Groundwater withdrawal using non-dedicated down-well pumps may be performed with a submersible pump or a bladder pump.

Electric submersible pumps provide an effective means for well purging and in some cases sample collection. Submersible pumps are particularly useful for situations where the depth to water table is greater than 20 feet and where the depth or diameter of the well requires that a large purge volume be removed before sample collection.

Commonly available submersible pumps include the Johnson-Keck pump model SP-82, the Grunfos Ready-Flow 2 pump, and disposable marine galley pumps, all of which are suited for operation in 2-inch or larger internal diameter wells.

Recently, the use of bladder pumps (positive gas-displacement pumps) has been promoted by the EPA for use in well purging and sampling primarily because the pumps can be operated at low flow rates (less than 1 liter per minute). Bladder pumps generally reduce the potential turbidity of the sample and theoretically reduce the potential for loss of VOC constituents, ultimately providing a more representative groundwater sample. Use of bladder pumps may require additional time for purging and sampling because of the low flow rate. Please note, however, that when using bladder pumps, it may not be necessary to purge an entire well volume of water prior to each check of the water quality parameters. Well purging is accomplished at such a low rate that, theoretically, the influent flow into the pump represents groundwater flow through the well screen, thereby eliminating the requirement for purging several entire well volumes of water before sample collection.

Bladder pumps usually consist of a stainless steel pump housing with an internal teflon or polyethylene bladder. Discharge tubing is generally made from teflon, polyethylene, or teflon-lined polyethylene. The pump is operated by lowering it into the water column within the well screen, then pulsing air into the bladder with an air compressor and pump controller unit. Pumps and controllers are often not interchangeable between manufacturers, therefore, it is usually necessary to have both items provided by the same manufacturer. Pump bladders are generally field-serviceable and replaceable.

A check of well condition may be required prior to inserting any down-well pump if the well has not been sampled for some time or if groundwater quality conditions are not known. The well condition check should include a check of casing plumbness as a bent well casing could cause a pump to get stuck. Casing plumbness can be checked by lowering a clean cylindrical tube with the approximate pump dimensions into the well. If the well casing is not plumb then an alternative purging method should be used.

The well inspection should also include a check of air quality or headspace conditions within the well for potentially explosive gasses and a check for free product which could foul the pump. Well casing headspace conditions can be monitored with a photoionization detector (PID) and/or an explosimeter for the presence of potentially explosive gasses. If potentially hazardous conditions exist, then an alternative purging method should be used. In general, it is rare for explosive conditions to be present.

The presence of free product should be determined before inserting the submersible pump into the well because free product may contaminate the pump's internal mechanisms making it extremely difficult to decontaminate.

An interface probe should be used to check for free product. Refer to Section 4.5 of this SOP for additional information on free product determination.

Electric Submersible Pump Procedure

Once the above well conditions have been assessed, and assuming its safe to proceed, slowly lower the submersible pump with attached discharge line into the monitoring well taking notice of any roughness or restriction within the well riser pipe. The pump should be placed in the uppermost section of the static water column of the monitoring well. The power cord should be attached to the discharge line with an inert material (i.e., zip-ties) to prevent the power cord from getting stuck between the pump, discharge line, and the well casing. Secure the discharge line and power cord to the well casing, using tape or a clamp, taking care not to crimp or cut either the discharge line or power cord.

Connect the power cord to the power source (i.e., rechargeable battery pack, auto battery, or generator) and turn the pump on. Voltage and amperage meter readings on the pump controller (if provided) should be monitored closely during purging. The operations manual for the specific pump used should be reviewed regarding changes in voltage/amperage and the potential impacts on pump integrity. Pumping should be discontinued if warning conditions occur and/or if the well is pumped to where drawdown falls below the pump's intake level.

If drawdown continues to the extent that the well is pumped dry, the pump should be shut off and the well allowed to recharge. This on/off cycle may be necessary in order to purge the well properly.

Measurements of the pumping rate, temperature, pH, and specific conductance (and/or other required parameters) should be made after each purge volume is removed and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been withdrawn and the field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

Bladder Pump Procedure

To operate the bladder pump system, the pump and discharge line should be lowered into the well close to the bottom of the well screen, then secured to the well casing with a clamp. The air compressor should then be turned on to activate pumping. The pump controller is used to vary the discharge rate to the required flow.

Measurements of the pumping rate, temperature, pH, and specific conductance (and/or other required parameters) should be made at periodic intervals while water is removed and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required field parameters have stabilized to within 10% of their preceding measurement. Generally, because of the low flow rate, samples are usually obtained from the bladder pump discharge line.

4.9 Sample Collection Methods and Procedures

4.9.1 Objectives

Groundwater samples can be collected using similar methods employed for purging, provided these methods do not adversely affect the quality of the groundwater. These methods include bailing, surface pumping and down-well pumping.

In most cases during sampling, groundwater will be transferred to the appropriate containers directly for the discharge source. During transfer, discharge tubing and other equipment shall not contact the inside of the sample containers. In addition, a clean pair of nitrile or latex gloves will be worn during sample collection and handling.

As a general rule of thumb, samples should be collected in order of decreasing volatilization of the target parameters. The preferred order of sample collection is as follows: volatile organic compounds, extractable organic compounds (e.g., semivolatile organic compounds, PCBs, pesticides), metals, and general water chemistry (ions and turbidity).

4.9.2 Bailers

The methods and procedures described in Section 4.9.2 also apply to collecting groundwater samples with a bailer. If a bailer was used to purge the well, the same bailer may be used for sampling. If other well purging equipment was used, a decontaminated or new disposable bailer should be used for sampling.

When volatile organic compounds are the target sampling parameter, a bottom discharge tip should be used during sample transfer. A discharge tip restricts the outflow of the sample from the bailer and diminishes the potential for volatilization. Reusable bailers may require a special screw-on tip fitted with a bottom discharge top. Disposable bottom discharge tips are usually supplied with disposable bailers.

Bailer cord shall be discarded after sampling is completed. Disposable bailers should only be used in one well. Reusable bailers should be appropriately decontaminated between uses.

4.9.3 Surface Pumps

The methods and procedures described in Section 4.9.3 for peristaltic and centrifugal pumps also apply to groundwater sample collection.

Peristaltic Pumps

Peristaltic pumps equipped with the appropriate type tubing will be used to collect groundwater from wells in which the water resides at a depth less than 20 feet. Sample bottles shall be filled directly from the pump's discharge line and care shall be taken to keep the discharge tube from contacting the sample container.

Groundwater samples requiring filtration prior to placement in sample containers can be placed in intermediate containers for subsequent filtration, or may be filtered directly with in-line disposable 0.45-micron filters, as described in SOP 7131.

After sampling is complete, all used tubing and filters shall be disposed of appropriately.

Centrifugal Pumps

Centrifugal pumps are generally not recommended for use in sample collection, especially when volatile organic compounds are the target analyte of interest. Samples for other analytes, however, may be obtained with use of an in-line sample trap. It is suggested that if samples cannot be obtained before going through the pump, that samples be obtained by using a bailer once purging is complete and pumping has ceased. Collecting samples from the pump discharge is not recommended.

After sampling is complete, all suction line tubing should be disposed of properly.

4.9.4 Down-Well Pumps

Electric Submersible Pump

Using the pump methods described in Section 4.9.4, groundwater samples can be collected directly from the pump discharge line, provided the discharge line is composed of inert material. Sample bottles will be filled directly from the discharge line of the pump. This method is generally not recommended for collection of volatile organic samples.

After sampling is complete, the pump, discharge line and power cord shall be decontaminated according to the procedures contained in SOP 7600 and/or disposed of as required by the project-specific work plan.

Bladder Pumps

Groundwater samples, including those collected for VOC analysis, may be collected directly from the pump discharge tubing under active pumping conditions. Sample bottles will be filled directly from the discharge line of the pump.

After sampling is complete, the pump, discharge line and power cord shall be decontaminated according to the procedures outlined in SOP 7600 and/or disposed of as required by the project-specific work plan.

4.10 Sample Filtration

The filtration of groundwater samples will be performed in accordance with SOP 7131. Groundwater samples collected for total dissolved metals analyses will be filtered prior to being placed in sample containers and properly preserved. Groundwater filtration will be performed using a peristaltic pump and a 0.45-micron in-line water filter. Disposable filters are commonly available in 0.45-micron size. Low-capacity or high-capacity cartridges are available and may be selectively used based on sample turbidity.

The filtration of groundwater samples shall be performed either directly from the pump discharge line or from laboratory-supplied intermediate containers. In either case, well purging shall be performed first. Fresh groundwater shall then be filtered directly into sample containers.

4.11 Sample Handling

All samples collected should be packaged and handled according to SOP 7510 and the project-specific sampling plan. Preservatives should be used where analytical methods require preservation. The QAPP will indicate the type of sample preservation necessary.

5.0 QUALITY CONTROL

5.1 Field Blank/Equipment Blank Sample Collection

Field blank samples serve as a quality assurance check of equipment and field conditions at the time of sampling. Field blank samples are usually prepared by transferring analyte-free water into a clean set of sample containers, then analyzing it as a sample. Sometimes, the analyte-free water is transferred over or through the sampling device before it is placed into the sample containers. This type of field blank sample is known as an equipment blank. The QAPP contains specific information regarding the type and number of field blanks or equipment blanks required for collection.

5.2 Field Duplicate Sample Collection

Field duplicate samples are collected for the purpose of providing two sets of results for comparison. These samples are used to assess precision. Duplicate samples are usually prepared by splitting the sample into two sets of sample containers, then analyzing each set as a separate sample. The QAPP contains specific information regarding the type and number of duplicate samples for collection.

5.3 MS/MSD Sample Collection

MS/MSDs provide information about the effect of the sample matrix on digestion and measurement methodology. For samples submitted for MS/MSD analysis, triple sample volume is generally required (contact the analytical laboratory for information specific to the project analytical parameters). The QAPP contains specific information regarding the frequency of MS/MSD samples.

6.0 DOCUMENTATION

Specific information regarding sample collection should be documented in several areas: the sample chain-of-custody record, sample collection record, field notebook, and sample labels, tags. Additional information regarding each form of documentation is presented in the following paragraphs:

6.1 Sample Chain-of-Custody Record

This ENSR standard form requires input of specific information regarding each collected sample for laboratory analytical purposes. The information requested includes site name and location, project number, field notebook reference, collection date and type of analysis requested. Each sample submitted for analysis is also listed individually using its field identification number, number and type of container, and requested analyses (see SOP 7510).

6.2 Groundwater Sample Collection Record

This form (Attachment 1 or 2) requires input of specific information regarding the collection of each individual sample including sample identification, water quality parameters, collection method, and containers/preservation requirements.

6.3 Field Logbook

This logbook should be dedicated to the project and should be used by field personnel to maintain a general log of activities throughout the sampling program. This logbook should be used in support of, and in combination with, the sample collection record. Documentation within the logbook should be thorough and sufficiently detailed to present a concise, descriptive history of the sample collection process.

6.4 Sample Labels/Tags

Sample labels shall be completed at the time each sample is collected and attached to each sample container. Labels will include the information listed below.

- Client or project name/project number
- Sample number
- Sample designation
- Analysis type
- Preservative
- Sample collection date
- Sample collection time
- Sampler's name

The project-specific work plan may also require the use of sample tags which generally contain the same information as the sample labels. Sample tags, if used, should be tied to each sample bottle with wire ties.

7.0 TRAINING/QUALIFICATIONS

Groundwater sample collection is a relatively involved procedure requiring formal training and a variety of equipment. It is recommended that initial sampling attempts be supervised by more experienced personnel. Sampling technicians should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous waste materials are considered to be present.

8.0 REFERENCES

EPA, Handbook for Sampling and Sample Preservation of Water and Wastewater, EPA-600/4-82-029, September 1982.

EPA, RCRA Groundwater Monitoring Technical Enforcement Guidance, November 1992.

Geotrans, Inc., RCRA Permit Writer's Manual, Groundwater Protection, prepared for the U.S. EPA, Contract No. 68-01-6464, October 1983.

Code of Federal Regulations, Chapter 40 (Section 261.4(d)).

Attachment 1 Ground Water Sample Collection Record
Attachment 2 Low Flow Ground Water Sample Collection Record

ENSR

Attachment 1
Groundwater Sample Collection Record

ENSR

Well/Piezo ID:

Ground Water Sample Collection Record

Client: ______ Date: ______
Project No: ______ Time: Start ______ am/pm
Site Location: ______ Finish ______ am/pm
Weather Conds: ______ Collector(s) ______

WATER LEVEL DATA: (measured from Top of Casing) Well ☐ Piezometer ☐

a. Total Well Length ______ c. Casing Material ______ e. Length of Water Column ______

b. Water Table Depth ______ d. Casing Diameter ______ f. Calculated Well Volume (see back) ______

WELL PURGING DATA

a. Purge Method ______

b. Acceptance Criteria defined (from workplan)
- Minimum Required Purge Volume (@ ______ well volumes) ______
- Maximum Allowable Turbidity ______ NTUs
- Stabilization of parameters ______ %

c. Field Testing Equipment Used: Make Model Serial Number

d. Field Testing Equipment Calibration Documentation Found in Field Notebook # ______ Page # ______

Time	Volume Removed (gal)	T° (C/F)	pH	Spec. Cond (umhos)	Turbidity (NTUs)	DO	Color	Odor	Other

e. Acceptance criteria pass/fail

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

If no or N/A - Explain below.

SAMPLE COLLECTION: Method: ______

Sample ID	Container Type	No. of Containers	Preservation	Analysis	Time

Comments ______

Signature ______ Date ______

ENSR

Attachment 2
Low Flow Ground Water Sample Collection Record

ENSR

Well ID:

Low Flow Ground Water Sample Collection Record

Client: ____________ Date: ____________ Time: Start ________ am/pm
Project No: ____________ Finish ________ am/pm
Site Location: ____________
Weather Conds: ____________ Collector(s): ____________

1. WATER LEVEL DATA: (measured from Top of Casing)

a. Total Well Length ________ c. Length of Water Column ________ (a-b) Casing Diameter/Material

b. Water Table Depth ________ d. Calculated System Volume (see back) ________

2. WELL PURGE DATA

a. Purge Method: ____________

b. Acceptance Criteria defined (see workplan)

- Temperature	3%	-D.O.	10%
- pH	± 1.0 unit	- ORP	± 10mV
- Sp. Cond.	3%	- Drawdown	< 0.3'

c. Field Testing Equipment used: Make Model Serial Number

Time (24hr)	Volume Removed (Liters)	Temp. (°C)	pH	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (feet)	Color/Odor

d. Acceptance criteria pass/fail

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

If no or N/A - Explain below.

(continued on back)

3. SAMPLE COLLECTION: Method: ____________

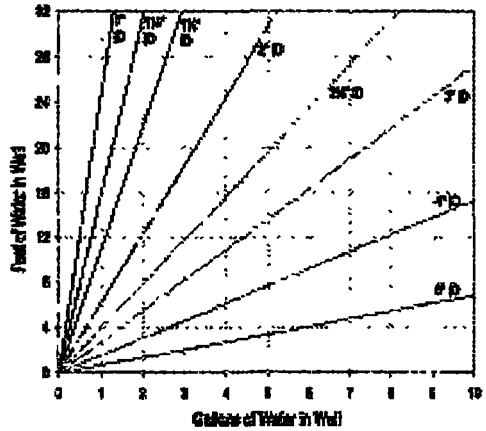
Sample ID	Container Type	No. of Containers	Preservation	Analysis Req.	Time

Comments ____________

Signature ____________ Date ____________

ENSR

Purge Volume Calculation



Volume / Linear Ft. of Pipe		
ID (in)	Gallon	Liter
0.25	0.0025	0.0097
0.375	0.0057	0.0217
0.5	0.0102	0.0386
0.75	0.0229	0.0869
1	0.0408	0.1544
1.25	0.0637	0.2413
1.5	0.0918	0.3475
2	0.1632	0.6178
2.5	0.2550	0.9653
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600

(continued from front)

Time (24 hr)	Volume Removed (Liters)	Temp (°C)	pH	Spec. Cond (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (ft)	Color/Odor

ENSR STANDARD OPERATING PROCEDURE

Number: 7510 **Date of Issue:** 2nd Qtr.1993

Revision: 2

Title: Packaging and Shipment of Samples

Organizational Acceptance	Authorization	Date
Originator	Christopher Carlio	3/13/84
Technical Reviewer	Arthur Lazarus	3/13/84
Technical Reviewer	Elaine Moore	3/13/84
Technical Reviewer		
Quality Assurance	Scott Whittemore	3/13/84

Revision #	Changes	Authorization	Date
1	• Chain-of-Custody procedure for hinged coolers added	Scott Whittemore Elaine Moore	9/19/86 10/13/86
	• Miscellaneous rewording		
2	• Format update	Mike Dobrowolski	4/27/93
	• Chain-of-Custody form update		

Organizational acceptance signatures are maintained on file with the original document in the Quality Assurance Library in Acton, MA.

SOP NO: 7510

ENSR ENSR Consulting and Engineering

Packaging and Shipment of Samples

Date: 2nd Qtr. 1993

Revision No: 2

Author: Christopher Carlio

Discipline: Geosciences

1.0 PURPOSE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the procedures associated with the packaging and shipment of samples. Two general categories of samples exist: environmental samples consisting of air, water and soil; and waste samples which include non-hazardous solid wastes and hazardous wastes as defined by 40 CFR Part 261.

2.0 RESPONSIBILITIES

2.1 Project Manager

It is the responsibility of the project manager to assure that the proper packaging and shipping techniques are utilized for each project.

2.2 Field Team Leader

The field team leader shall be responsible for the enactment and completion of the packaging and shipping requirements outlined in the project specific sampling plan. The field team leader shall be responsible to research, identify and follow all applicable U.S. Department of Transportation (DOT) regulations regarding shipment of materials classified as waste.

3.0 REQUIRED MATERIALS

- Sample cooler
- Bubble wrap
- "Blue Ice" refreezable ice packs
- Fiber tape

- Zip lock plastic bags

4.0 METHOD

The objective of sample packaging and shipping protocol is to identify standard procedures which will minimize the potential for sample spillage or leakage and maintain field sampling program compliance with U.S. EPA and U.S. DOT regulations.

The extent and nature of sample containerization will be governed by the type of sample, and the most reasonable projection of the sample's hazardous nature and constituents. The EPA regulations (40 CFR Section 261.4(d)) specify that samples of solid waste, water, soil or air, collected for the sole purpose of testing, are exempt from regulation under the Resource Conservation and Recovery Act (RCRA) when all of the following conditions are applicable:

- Samples are being transported to a laboratory for analysis;
- Samples are being transported to the collector from the laboratory after analysis;
- Samples are being stored (1) by the collector prior to shipment for analyses, (2) by the analytical laboratory prior to analyses, (3) by the analytical laboratory after testing but prior to return of sample to the collector or pending the conclusion of a court case.

Qualification for transportation as described above require that sample collectors comply with U.S. DOT and U.S. Postal Service (USPS) regulations. If U.S. DOT and USPS regulations are found not to apply, the following information must accompany all samples and will be entered on a sample specific basis on chain of custody records:

- sample collector's name, mailing address and telephone number,
- analytical laboratory's name, mailing address and telephone number,
- quantity of sample,
- date of shipment,
- description of sample, and

In addition, all samples must be packaged so that they do not leak, spill or vaporize.

4.1 Place plastic bubble wrap matting over the base and bottom corners of each cooler or shipping container as needed to manifest each sample.

4.2 Obtain a chain of custody record as shown in Figure 1 and enter all the appropriate information as discussed above. Chain of custody records will include complete information for each sample. One or more chain of custody records shall be completed for each cooler or shipping container as needed to manifest each sample.

4.3 Wrap each sample bottle individually and place standing upright on the base of the appropriate cooler, taking care to leave room for some packing material and ice or equivalent. Rubber bands or tape should be used to secure wrapping, completely around each sample bottle.

4.4 Place additional bubble wrap and/or styrofoam pellet packing material throughout the voids between sample containers within each cooler.

4.5 Place ice or cold packs in heavy duty zip-lock type plastic bags, close the bags, and distribute such packages over the top of the samples. Add additional bubble wrap/styrofoam pellets or other packing materials to fill the balance of the cooler or container.

4.6 Obtain two pieces of chain of custody tape as shown in Figure 2 and enter the custody tape numbers in the appropriate place on the chain of custody form. Sign and date the chain of custody tape.

4.7 To complete the chain of custody form enter the type of analysis required for each sample, by container, under the "ANALYSES" section. Under the specific analysis enter the quantity/volume of sample collected for each corresponding analysis.

4.8 If shipping the samples where travel by air or other public transportation is to be undertaken, sign the chain of custody record thereby relinquishing custody of the samples. Relinquishing custody should only be performed when directly transmitting custody to a receiving party or when transmitting to a shipper for subsequent receipt by the analytical laboratory. Shippers should not be asked to sign chain of custody records.

4.9 Remove the last copy from the chain of custody record and retain with other field notes. Place the original and remaining copies in a zip-lock type plastic bag and place the bag on the top of the contents within the cooler or shipping container.

4.10 Close the top or lid of the cooler or shipping container and with another person rotate/shake the container to verify that the contents are packed so that they do not move. Improve the packaging if needed and reclose.

4.11 Place the chain of custody tape at two different locations on the cooler or container lid and overlap with transparent packaging tape. For coolers with hinged covers, if the hinges are attached with screws, chain of custody tape should also be used on the hinge side.

4.12 Packaging tape should be placed entirely around the sample shipment containers. A minimum of two full wraps of packaging tape will be placed at least two places on the cooler. Shake the cooler again to verify that the sample containers are well packed.

4.13 When transporting samples by automobile to the laboratory, and where periodic changes of ice are required, the cooler should only be temporarily closed so that reopening is simple. In these cases, chain of custody will be maintained by the person transporting the sample and chain of custody tape need not be used. If the cooler is to be left unattended, then chain of custody procedures should be enacted.

4.14 If shipment is required, transport the cooler to an overnight express package terminal or arrange for pickup. Obtain copies of all shipment records as provided by the shipper.

4.15 If the samples are to travel as luggage, check with regular baggage.

4.16 Upon receipt of the samples, the analytical laboratory will open the cooler or shipping container and will sign "received by laboratory" on each chain of custody form. The laboratory will verify that the chain of custody tape has not been broken previously and that the chain of custody tape number corresponds with the number on the chain of custody record. The analytical laboratory will then forward the back copy of the chain of custody record to the sample collector to indicate that sample transmittal is complete.

5.0 QUALITY CONTROL

Not Applicable

6.0 DOCUMENTATION

As discussed in Section 4.0 the documentation for supporting the sample packaging and shipping will consist of chain of custody records and shipper's records. In addition a description of sample packaging procedures will be written in the Field Log Book. All documentation will be retained in the project files following project completion.

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CHAIN OF CUSTODY RECORD

Page ___ of ___

Client/Project Name	Project Location
Project Number:	Field Logbook No :
Sampler (Print Name) /Affiliation	Chain of Custody Tape No.:
Signature	Send Results/Report to

Field Sample No./ Identification	Date	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered	Analysis Requested						Lab I.D.	Remarks

Relinquished by (Print Name)	Date:	Received by (Print Name)	Date:	Analytical Laboratory (Destination):
Signature	Time:	Signature:	Time:	
Relinquished by (Print Name)	Date:	Received by (Print Name)	Date:	
Signature	Time	Signature:	Time:	
Relinquished by (Print Name)	Date	Received by (Print Name)	Date	
Signature	Time	Signature:	Time	Serial No. 6118

Date ____________________

Sig. ____________________

Nº 002233

B

STL Denver

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Controlled Copy No. ______________

Implementation Date 10-30-00

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: BUILDING SECURITY

(SUPERSEDES: NONE)

Prepared by: Rhonda Johnson and Larry Penfold

Reviewed by: 10-27-00
Technical Specialist, Randy Rakowski

Approved by: 10/27/00
Quality Assurance Manager, Larry Penfold

Approved by: 10/27/00
Environmental Health and Safety Coordinator, Scott Kelly

Approved by: 10/27/00
Laboratory Director, Timothy M. O'Shields

1. PURPOSE AND SCOPE

The purpose of this SOP is to provide instructions for maintaining building security. Building security is necessary to protect company assets, protect the confidentiality of samples and client information, and is an important element in legal defensibility of the data we produce.

STL Denver's facility security includes controlled access to the building, sign-in and badging requirements for all visitors, and active electronic security during non-working hours.

2. RESPONSIBILITIES

2.1. The EH&S Coordinator conducts training for new employees on this SOP.

2.2. The Human Resource Coordinator is responsible for issuing employee identification numbers and conducting hands-on training with the hand scanner security devices.

2.3. The receptionist is responsible for issuing visitor badges and for visitor sign-in during normal business hours.

2.4. Employees escorting visitors are responsible for ensuring that visitation procedures are followed and that data confidentiality has not been compromised.

2.5. The facility maintenance staff is responsible for maintaining the alarm system.

2.6. Our security company is responsible for notifying the designated parties if an entry or fire alarm is detected.

3. SAFETY

New employee and visitor safety orientation are described in the Chemical Hygiene Plan.

4. PROCEDURE

4.1. All exterior doors to the facility will remain locked at all times, with the exception of the front entrance during the hours 08:00 to 17:15, and the roll-up receiving doors when deliveries are being made.

4.2. There are two entrance doors for regular use by employees. There are double entries with two key coded security devices at each of the two entrances. One is an interior combination key pad / hand scan that is operational at all hours. All employees must enter a key code and hand scan when arriving or leaving the premises. The other is an exterior key pad system that requires an additional key code for employees to open the exterior door.

4.2.1. Front (Southeast) Entrance

During the hours of 08:00 to 17:15, the main (southeast) entrance and the reception areas are controlled by the receptionist. The alarm system is not activated during this time period.

4.2.2. Rear (Northwest) Entrance

There is no receptionist at this entrance. The interior key pad / hand scan must always be used. The exterior security device must be used outside the hours of 08:00 to 17:15.

4.3. The roll-up doors in the courtyard of the building are for shipping and receiving only. The exterior key pad for the roll-up doors is disabled during non-working hours, which completely prevents the use of these doors.

4.4. Other doors are for emergency exits only. Do not use any emergency to exit or enter the building during non-emergencies.

4.5. Use of the Hand Scanners

When the building alarm is active (17:15 to 08:00), entry to the building or exit from the building must be made through the front (southeast) or rear (northwest) entrances where hand scanners are located. These hand scanners allow entrance or exit, temporarily disabling the building alarm system at the exit/entry location. The alarm is only deactivated for 15 seconds. The doors must be closed within 15 second or the alarm will be activated. All other exits will remain armed during exit/entry from main entrance or northwest entrance.

4.6. In the event of an unauthorized entry, our security company (Central Security) will call the local police department to investigate. In the event of a fire alarm, the local fire department will be notified.

4.7. If the alarm is accidentally activated the following steps need to be taken:

4.7.1. Proceed to one of the main entrances.

4.7.2. Key in the reset code on the keypad located above the hand scanner. The reset code is 24361#. If you are a temporary employee, get a permanent employee immediately.

4.7.3. Call Central Security at 303-825-8195. Only call to have the alarm reset when if it is definitely verified that it is a false alarm.

4.7.4. Indicate to the operator at Central Security that this is a call for a false alarm from STL Denver at 4955 Yarrow in Arvada, CO. Give the operator our account number. The account number is 57462.

4.7.5. The operator will ask for a code. The code is the last four numbers of each employee's Social Security number. Give the code to the operator.

4.7.6. If Central Security calls before an employee is able to call Central Security, give the same information listed in 4.4.4 and 4.4.5.

4.7.7. Central Security may call again to verify a false alarm.

4.5 If there is a problem with deactivating the alarm during a false alarm, contact one of the individuals listed in Table 1 for what actions to take.

Table 1

Contact Phone Numbers and Reset Code

Reset Code	24361#
Central Security	303-825-8195
Account number	57462
Code	Last four digits of permanent employees Social Security number
Scott Kelly	303-432-8421
Randy Rakowski	303-431-7020 303-589-0383 (cell phone)
William Rhoades	303-655-7421

STL Denver

SOP No: CORP-QA-0010DEN
Revision No: 3
Revision Date: 10/08/01

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Implementation Date 10/08/01

STL DENVER STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION SYSTEM

(SUPERSEDES: CORP-QA-0010 Revision 2)

Prepared By:	Larry Penfold	
Reviewed By:	Rhonda Johnson, Technical Specialist	
Approved By:	Larry Penfold, Quality Assurance Manager	10/8/01
Approved By:	Scott Kelly, Environmental, Health and Safety Coordinator	10/8/01
Approved By:	Timothy O'Shields, Laboratory Director	10/8/01

1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The STL Quality Assurance Management Plan (QMP) and STL Denver Laboratory Quality Manual (LQM) require documentation of instances of deviations from established control limits, approved standard operating procedure (SOPs), or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from STL Denver policies and procedures and documented client specifications including root causes and corrective actions taken to remedy the nonconformance. The NCM will be stored in electronic form in a database called Clouseau, with any supporting material filed in QA as necessary.

1.2. This document applies to procedures, services, analytical data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. The system used to handle the sample receiving and client-related issues, including holding time violations (HTVs), emphasizes the <u>immediate</u> <u>notification</u> of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Nonconformances can be identified by associates in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through reviews of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of STL Denver's product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.

2.2. **Group Leader / Supervisor:** Each supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors review hardcopy or electronic versions of NCMs and forward them to the appropriate project manager and quality assurance personnel. Supervisors are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The project manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The project manager communicates conformance problems to clients and documents decisions made with clients. The project manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The project manager may withhold final reports to clients until corrective actions agreed to with the client have been completed. Project managers are also responsible for initiating an NCM for client complaints or inquiries.

2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for reviewing all NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and spot audits conducted to ensure that long-term corrective actions have been successfully completed. Significant conformance issues are included in the Monthly QA Report to Management, which is distributed to the Laboratory Director, Regional Manager, and Corporate QA Director. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations Manager** : The Operations Manager shall ensure that corrective actions are correct and have been implemented. The operations manager review and concurrence shall be documented in the database as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Director:** The Laboratory Manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Procedures shall be carried out in a manner that protects the health and safety of all associates.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported **immediately** to a laboratory supervisor.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to initiate an electronic nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or standard materials deviate from established specifications. An NCM may also be initiated to document and observation noted during the course of the analysis that may or may not have an effect on the resulting data (e.g., unusual color or odor). All nonconformances with the exception of matrix-related failures require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions). ALL holding time violations (HTVs) require an NCM.

4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPjPs Quality Assurance Summaries, or equivalent systems). Any intentional deviation from an SOP must be pre-approved by the QA manager and Supervisor. Any deviation from an SOP or client requirement not previously approved must be documented using the NCM process.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple lot numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues. If the nonconformance involves projects for multiple project managers, then the NCM will need to be routed to each project manager.

4.2. How to Process the NCM using Clouseau (SQL version)

4.2.1. Initiating the NCM

4.2.1.1. While properly logged into a PC where Clouseau has been installed, start the Clouseau program.

4.2.1.2. At the Main Menu, select New.

4.2.1.3. Enter the information as prompted by the Clouseau program. At a minimum select the Production Area, Category (anomaly, deficiency or observation - observations are not reviewed by supervisors or QA), NCM Type, and NCM Description.

4.2.1.4. Select whether the nonconformance affects entire lots, entire batch, specific sample, specific analysis (work order) or other. Other should only be used in cases such as client complaints or instrument tag out, that do not affect specific samples. If using one of the "entire" options, extra samples may be deleted by highlighting and using the delete key on the keyboard. The view may be resorted by clicking on one of the headers.

4.2.1.5. Complete the Comments/Details and Corrective Action sections of the Create NCM Form. These sections may be filled out jointly with the supervisor. Consult the Project Manager or the Operations Manager and QA Manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.2.1.5.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples.

4.2.1.5.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.2.1.6. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff before the NCM is considered closed.

4.2.1.7. Save the NCM using the appropriate command in Clouseau. When presented with the Send E-Mail form, the supervisor of the affected Production Area will be listed in the "Notify Now" box, while affected Project Managers and QA staff will be listed in the "Notify

Later" box. Verify that these names are correct. If any personnel should be informed that are not listed, add their names to the "Notify Now" box by double-clicking on their names in the Personnel box.

4.2.1.8. Initiate the NCM notification process by selecting SEND EMAIL.

4.2.2. Supervisory Review and Approval

4.2.2.1. The supervisor will receive notification of the NCM via e-mail. The supervisor must log into a computer workstation where Clouseau has been installed, and run Clouseau.

4.2.2.2. Using the Review NCM form, select the NCM to be reviewed and highlight.

4.2.2.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. If the corrective action has not been determined, the situation must be referred to the project manager and the operations manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations manager needs to be made aware of the issues, the QA staff will notify the operations manager using the Under Review/Send Email options of Clouseau.

4.2.2.4. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired. Refer to Section 4.2.4.8.

4.2.2.5. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. The supervisor selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process, which will now route the NCM to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.2.3. Project Manager Review, Client Notification, and Project Documentation

4.2.3.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate using the Client Notification Form in Clouseau the date and method of notification and client's response .

4.2.3.2. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. Notify the supervisor of any changes made to the corrective action plan.

4.2.3.3. The project manager must select the "PM" button and document how client notification was done, and what, if any, response was made. If multiple projects are associated with one NCM, each project must be highlighted in this screen, and appropriate notification documented.

4.2.3.4. The project manager selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing to the QA office. The QA office then must review the NCM in a timely manner, generally within 72 hours.

4.2.3.5. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has approved the NCM. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned into the QA staff if corrections are needed in the database (QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.2.4. Quality Assurance Review and Trending

4.2.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices and policies.

4.2.4.2. NCMs will be reviewed to ensure that the corrective action was completed and effective in addressing the root cause of the nonconformity to prevent recurrence.

4.2.4.3. Clouseau's reporting and tracking system will be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.2.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. As an initial investigation step for quality control issues, control charts are frequently generated from the TraQAr Control Limits program. Recurrent the appropriate lab group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area along with supporting evidence.

4.2.4.5. The QA staff shall conduct spot follow-up assessments to confirm that correction of systematic problems is successful. These assessments are done on at least an annual basis.

4.2.4.6. The approval of the QA manager or designee is required in Clouseau to indicate that the NCM has been closed.

4.2.4.7. The Clouseau database comprises the central file/record of all closed NCMs.

4.2.4.8. Instrument/Equipment Nonconformance Tag

4.2.4.8.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a clearly visible tag or sign indicating the nonconforming condition.

4.2.4.8.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM. Identify the instrument by name and/or identification number and briefly describe the problem.

4.2.4.8.3. Upon saving the NCM, the NCM number indicated by Clouseau shall be recorded in the instrument's maintenance log.

4.2.4.8.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record the back to control information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

5. DEFINITIONS

5.1. ***Nonconformance:*** an unplanned deviation from an established protocol or plan. The deviation may be the result of STL Denver's actions, then termed a **deficiency**, or the result of events beyond the control of STL Denver, then termed an **anomaly**. An **observation** is any phenomena that may affect data quality and so needs to be included in the final report case narrative.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrates a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects must be documented, but an NCM is not required. A procedure is not performed as described in the applicable SOP or QA Policy, ***except*** in cases where the procedure has been performed according to a client-specified document STL Denver has agreed to follow (e.g., EPA SOWs and QAPjPs).

5.1.2. A practice or procedure is not performed as described according to a client or project document that STL Denver has agreed to follow.

5.1.3. Purchased materials or services are determined to be defective and their use would affect data quality.

5.1.4. ANY holding time violations (HTVs) occurs regardless of what or whose actions caused them including ACTS of GOD. Information in the Clouseau database is used by QA staff to generate a weekly HTV Report to local laboratory management.

5.1.5. Any lab communication (such as description of sample or sample performance) from the analyst to supervisor, PM, or QA.

5.1.6. A formal NCM is not required for routine minor instrument maintenance, malfunctions, and power failures which can be documented in instrument maintenance logbooks.

5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.

5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.

5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Reference Documents

6.1.1. STL Denver Laboratory Quality Manual (LQM), current revision.

6.1.2. STL Quality Management Plan (QMP), current revision

6.1.2. STL Quality Management Plan (QMP), current revision

6.1.3. ANSI/ASME NQA-1, Chapter II, Basic Requirement 15 "Control of Nonconforming Items." Supplement 15S-1 "Supplementary Requirements for the Control of Nonconforming Items."

6.1.4. NELAC Quality Standards, Section 5.5.3.5; July 1999.

6.2. Changes From Previous Version

6.2.1. Company name changed from Quanterra to STL

6.2.2. Job titles for responsible parties changed to current titles.

6.2.3. Table of contents was removed because this is a short SOP.

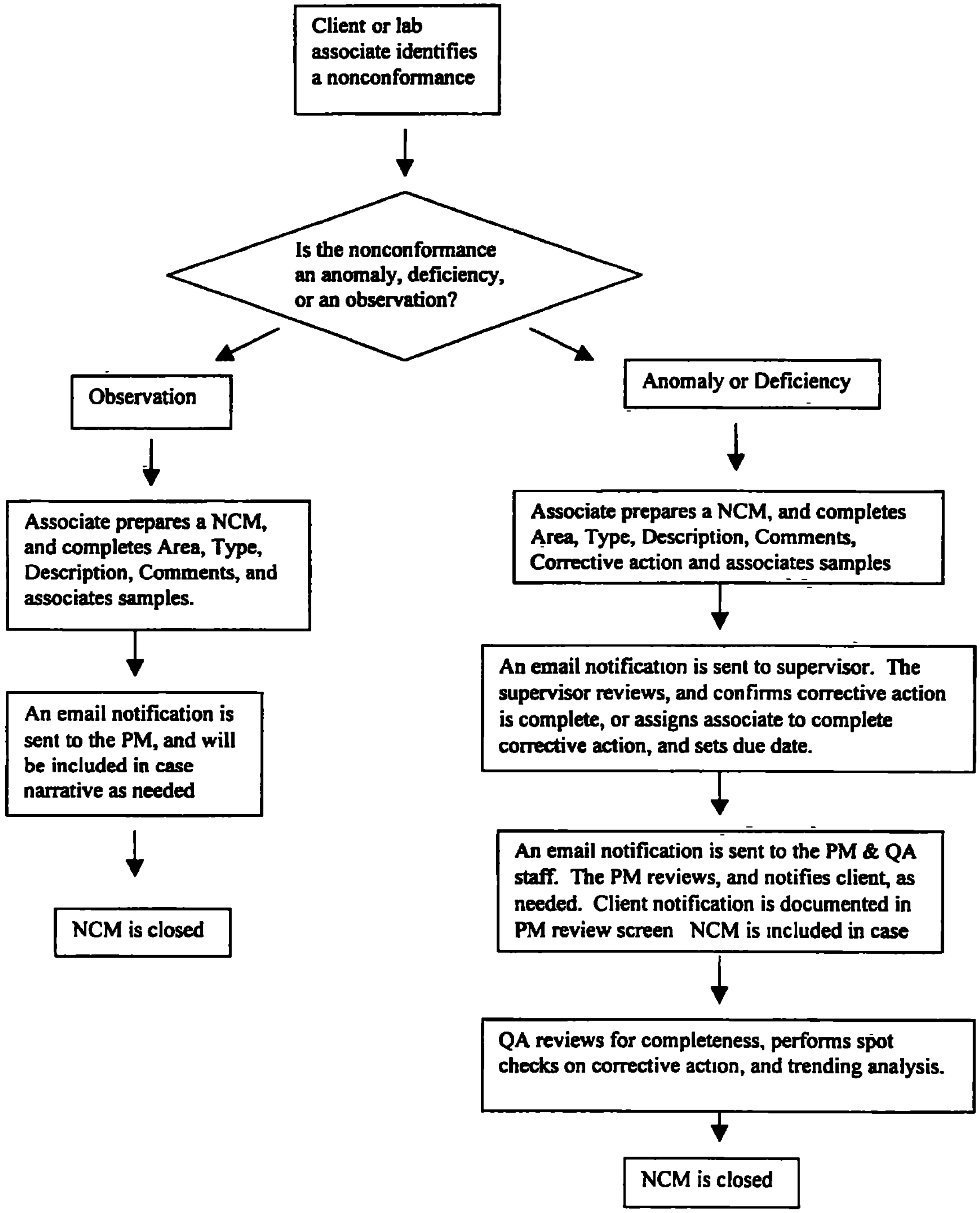
6.2.4. The documentation in Clouseau of "observations" is discussed.

6.2.5. Details about the 2001 SQL version of Clouseau are added.

6.3. Appendices

6.3.1. Attachment A: Nonconformance Memo Generated from Clouseau

Flow Chart for Internal NCM



ATTACHMENT A
EXAMPLE LABORATORY NCM FROM CLOUSEAU PROGRAM

Clouseau
Nonconformance Memo

SEVERN TRENT SERVICES

NCM # **04-14936**
NCM Initiated By. Blake Haworth
Date Opened 10/05/2001
Date Closed. 10/08/2001

Classification **Anomaly**
Status· **CLOSED**
Production Area GC-SV
Tests 614, 8141A
Lot #'s (Sample #'s) D1I250236 (1), D1I250257 (1,2,3,4,5), D1I250299 (2), D1I260135 (1,2), D1I260137 (1), D1I260169 (1,10,2,3), D1I260260 (4), D1I260311 (2,3), D1I270321 (2), D1I280248 (1), D1J010000 (154),
QC Batches. 1274154

Nonconformance· Matrix QC failure (matrix interference uncertain)
Subcategory· MS/MSD recovery outside of control limits

Problem Description / Root Cause

Name	Date	Description
Blake Haworth	10/05/2001	The matrix spike and/or matrix spike duplicate recoveries were outside of established limits. This also caused the RPDs out of the control limits for those compounds The LCS and blank were in control It is not obvious whether or not this is due to a matrix interference Compounds effected Phorate Total Demton Diazinon

Corrective Action

Name	Date	Corrective Action
Blake Haworth	10/05/2001	Report samples with a case narrative.

Client Notification Summary

Client	Project Manager	Notified	Response	How Notified	Note

Response	Response Note

Quality Assurance Verification

Verified By	Due Date	Status	Notes
PENFOLDL		Verification not required or requested	

Approval History

Date Approved	Approved By	Position

10/05/01 HAWORTHB
10/05/01 YODERK
10/08/01 DERUZZOG
10/08/01 PENFOLDL

STL Denver

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Implementation Date 10/29/01

STANDARD OPERATING PROCEDURE
TITLE: POLYNUCLEAR AROMATIC HYDROCARBONS BY SELECTIVE ION MONITORING FOR CITY OF ST. LOUIS PARK
(SUPERSEDES: DEN-MS-0005 REV. 0)

Prepared by: Tim O'Donnell

Reviewed by: [signature] 10/29/01
Technical Specialist, Michael Klasner

Approved by: [signature] 10/26/01
Quality Assurance Manager, Larry Penfold

Approved by: [signature] 10/26/01
Environmental Health and Safety Coordinator, Scott Kelly

Approved by: [signature] 10/29/01
Laboratory Director, Timothy M. O'Shields

Received OCT 29 2001 Tim O'Shields

1. SCOPE AND APPLICATION

This procedure is a Gas Chromatography/Mass Spectrometry (GC/MS) technique developed for the purposes of measuring polynuclear aromatic hydrocarbons (PAH) at the part per trillion (ppt, ng/L) level. The analyte list for which this method applies is attached. This method should be used for the analysis of water samples previously characterized by a method such as 8270C and containing organic components at less than 10,000 ng/L. Samples containing greater than 10,000 ng/L semivolatile organics should be analyzed by a method designed to detect at higher (ppb) levels.

2. SUMMARY OF METHOD

2.1. This method has been designed for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion level (ppt,ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The compounds listed in Table I can be quantitatively determined using this analytical method.

2.2. This method has three options for the extraction of the samples depending on the sample type. The three options include the low level, the ppt 75 level, and the medium level extraction. The low level and the ppt 75 level options have typical reporting limits of 1.0 ppt. The ppt 75 level includes a higher surrogate and spike level to accommodate dilutions. The medium level option is eighty times higher in detection limits. A volume of sample dependent of the extraction option chosen is extracted with methylene chloride. Analysis of the concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions.

3. DEFINITIONS

3.1. Refer to the LQM for QA/QC definitions of terms used in this document.

3.2. Selected Ion Monitoring - A mass spectrometry technique that provides lower detection level capability.

3.3. Primary Ion Area - The signal chosen for quantitation purposes.

3.4. Secondary Ion Area - The signal chosen for identification and confirmation purposes.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

4.2. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

4.3. An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which contains the quantitation mass ion. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not the specified limits, then interferences may be present.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates. The following requirements must be met: Eye protection that satisfies ANSI Z87.1 (as per the STL Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. Refer to the STL Corporate Safety Manual for a complete description of personal protection equipment. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information is available and must be read from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.2. Chemicals known to be **flammable** are:
Acetone, methanol, and toluene. Although the PAH compounds used for this test are flammable, they are used diluted in solvent for this test. The solvent may be flammable.

5.3. Chemicals that have been classified as **carcinogens**, or **potential carcinogens**, under OSHA include:

Methylene chloride, benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluoranthene, benzo(j)fluoranthene, benzo(g,h,i)perylene, chrysene, dibenz(a,h)anthracene, indeno(1,2,3)pyrene, and quinoline.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware

6.1.1. Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, and finally with acetone.

6.1.2. Glassware should **not** be oven dried or heated in a muffle furnace at 400°C for 15 to 30 minutes. Successive solvent rinses of the CLLE, separatory funnel and K/D glassware are required to minimize low level contamination of samples. Each glassware set is pre-extracted using blank reagent water and full solvent volumes. This pre-extraction step is performed using the same amount of time allotted for the sample batch extraction. The reagent water that has been pre-extracted is then used for preparing the batch method blank and laboratory control spikes.

6.1.3. Store glassware inverted or in sealed containers capped with aluminum foil. The use of high purity reagents and solvents helps to minimize interference problems.

6.2. Separatory funnel - 4000 mL with Teflon stopcock or continuous liquid-liquid extractor, 2000 mL or 4000 mL with a condenser.

6.3. Drying column - glass funnel with about 10 cm anhydrous sodium sulfate.

6.4. Concentrator tube, Kuderna-Danish - 10 mL, graduated with separate N-Evap tubes (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Aluminum foil is used to prevent evaporation of extracts.

6.5. Snyder column, Kuderna-Danish - Three-ball macro (Kontes K-503000-0121 or equivalent).

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Environmental Health and Safety Coordinator, Scott Kelly

Approved by: 10/26/01
Laboratory Director, Timothy M. O'Shields

Received
OCT 29 2001
Tim O'Shields

6.6. Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs or clips.

6.7. Nitrogen evaporation device equipped with a water bath that can be maintained at 30-35°C. The N-Evap by Organomation Associates, Inc., South Berlin, MA (or equivalent) is suitable.

6.8. Micro reaction vessels, 1.8 mL vials with Teflon caps.

6.9. Gas Chromatograph

The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

6.10. A DB-625.5 30 meter fused silica capillary column, 0.5 mm film thickness, or equivalent.

6.11. Mass Spectrometer

6.11.1. A mass spectrometer operating at 70 eV (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

6.11.2. A computer system interfaced to the mass spectrometer allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

7. REAGENTS AND STANDARDS

7.1. Reagent water

Reagent water is defined as water in which the target compounds are not observed at or above the method detection limit. Reagent water used for this method is pre-extracted (see section 6.1.2 for details).

7.2. Acetone, distilled in glass, or equivalent.

7.3. Methanol, distilled in glass, or equivalent.

7.4. Methylene chloride, distilled in glass, or equivalent.

7.5. Sodium sulfate

(ACS) Granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray.

7.6. Surrogate Spiking Solutions

Depending on the extraction option chosen low, low 75, or medium a surrogate solution is made by diluting stock surrogate solutions from a vendor. The compounds in the surrogate solutions are naphthalene-d_8, fluorene-d_{10}, and chrysene-d_{12}. The low level surrogate spike solution is at 10 ng/mL. 1.0 mL of the surrogate spike solution is added to 2.0 L of sample. The ppt 75 level surrogate spike solution is at 150 ng/mL. 1.0 mL of the surrogate spike solution is added to 2.0 L of sample. The medium level surrogate spike solution is at 2000 ng/mL. 1.0 mL of the surrogate spike solution is added to 0.5 L of sample.

7.7. Internal Standard Solutions

A solution containing 6000 ng/mL is prepared from vendor stock solutions.

7.8. Matrix Recovery Standard Spiking Solution

A solution containing the following compounds at the listed concentrations is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone. The concentrations of the spiking solution for the low, ppt 75 and medium level extractions are shown below:

Compound	Low spiking Solution (ng/mL)	Medium spiking Solution (ng/mL)	Low 75 spiking Solution (ng/mL)
Naphthalene	40	2000	300
Fluorene	40	2000	300
Chrysene	40	2000	300
Indene	40	2000	300
Quinoline	40	2000	300
Benzo(e)pyrene	40	2000	300
2-methylnaphthalene	40	2000	300

The low level matrix spike solution is at 40 ng/mL. 1.0 mL of the surrogate spike solution is added to 4.0 L of sample. The ppt 75 level matrix spike solution is at 300 ng/mL. 1.0 mL of the surrogate spike solution is added to 4.0 L of sample. The medium level surrogate spike solution is at 2000 ng/mL. 1.0 mL of the surrogate spike solution is added to 0.5 L of sample.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The samples are collected into four 1-liter or one 1-gallon amber glass containers chilled to 4°C ± 2°C and shipped via over-night carrier to the laboratory.

8.2. The samples must be protected from light and refrigerated at 4°C ± 2°C from the time of receipt until extraction and analysis. After analysis, extracts and unused sample volume must be protected from light and refrigerated at 4°C ± 2°C.

8.3. Samples must be extracted within 5 days of the time of sample receipt. Samples are required to be shipped the same day samples are collected using an overnight carrier.

8.4. Extracts must be analyzed within 40 days from sample extraction.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability & MDL Study. See section 13 of this SOP for details concerning MDL and IDC studies.

9.2. An initial demonstration of capability (IDC) and a method detection limit study must be performed before samples can be analyzed.

9.3. Additional Compounds

This method is capable of monitoring many compounds other than those listed in the tables below. Prior to reporting an additional compound by this method, a validation procedure for the compound should be performed. It should be noted in the final report if validation procedures have not been performed for an analyte

The Quality Control measures defined for this method are summarized in Table 9-1. See also QC Policy (QA-003) for details.

TABLE 9-1

QC Type	Frequency
Internal Standards	each sample
Surrogate	each sample
Matrix spike/spike duplicate	1 set per analytical batch or every 20 samples - which ever is most frequent
Laboratory Control Sample	1 per analytical batch or every 20 samples - which ever is most frequent
Method Blank	1 per analytical batch

9.4. Method Blank

9.4.1. Laboratory reagent water must be treated by extracting with methylene chloride prior to use as method reagent water for this method.

9.4.2. A method blank is analyzed with each analytical batch not exceeding 20 samples.

9.4.3. Due to the low-level nature of this analysis, low level blank contamination of target analytes is routinely encountered. Target analytes that are detected above the reporting limit in the blank must be flagged on any associated sample's report with a "B" qualifier. If the method blank contains any of the carcinogenic PAHs at concentrations greater than the method detection limit (MDL), or any other target PAH compound at a concentration 5 times greater than the MDL, the method blank will be considered out of control. Corrective action will include reanalysis of the blank extract, an investigation into laboratory sources of contamination and qualifying that sample data relates to the blank. Blank level contamination should be considered the minimum level of contamination in all samples that are analyzed with the blanks.

9.5. Matrix Spike and Spike Duplicate Analyses

9.5.1. Samples designated for matrix spike analysis are spiked as described in section 7.8.

9.5.2. The laboratory will perform a matrix spike/ spike duplicate pair of QC samples for each analytical batch not exceeding 20 samples.

9.5.3. The initial matrix spike criteria are as follows:

Spike Component	Acceptance Criteria
1H- Indene	20-150
Naphthalene	20-150
Quinoline	20-150
2- Methylnaphthalene	20-150
Fluorene	20-132
Chrysene	20-132
Benzo(e)pyrene	20-150

One compound is allowed to be below the above acceptance criteria. The average recovery for the spike pair must also fall into the above criteria with one compound being allowed below the acceptance criteria.

9.5.4. Matrix spike compound criteria will be developed as results are collected and will be updated annually.

9.5.5. If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, then the reanalysis data will be used. If not, the data for the sample will be reported but qualified as being outside the acceptance criteria of the method. Both the original and reanalysis data will be reported.

9.6. Laboratory Control Samples

9.6.1. A Laboratory Control Sample (LCS) is analyzed with each analytical batch not exceeding 20 samples.

9.6.2. The LCS results are compared to established spike recovery limits.

9.6.3. One DCS pair may be required for specific projects.

9.7. Internal Standards

The internal standards are monitored in all field samples and QC samples. The internals standards areas should be between 50% and 200% of the continuing calibration standard area.

9.8. Surrogate Compound Analysis

9.8.1. The laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compounds will be spiked into the sample prior to extraction and will measure individual sample matrix effects associated with sample preparation and analysis. Surrogates will include naphthalene-d_8, fluorene-d_{10}, and chrysene-d_{12}.

9.8.2. STL Denver will take corrective action whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

Surrogate	Acceptance Criteria % Low-Level
Naphthalene-d_8	21-108
Fluorene-d_{10}	41-162
Chrysene-d_{12}	10-118

9.8.3. Corrective Action

9.8.3.1. Check calculations to assure there are no errors;

9.8.3.2. Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;

9.8.3.3. If the upper control limit is exceeded for only one surrogate, and the instrument calibration, surrogate standard concentration, etc. are in control, it can be concluded that an interference specific to the surrogate was present that resulted in high recovery and this interference would not affect the quantitation of other target compounds.

9.8.3.4. If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded as not calculated (NC).

9.8.3.5. Reanalyze the sample or extract if the steps above fail to reveal a problem. If reanalysis of the extract yields surrogate recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.

10. CALIBRATION AND STANDARDIZATION

10.1. The GC/MS is not be tuned to meet decafluorotriphenylphosphine (DFTPP) ion abundance criteria. This requirement is not appropriate for selected ion monitoring (SIM) methods. The analyst should tune the instrument to maximize the sensitivity for the compounds being analyzed as described below.

10.2. Mass tuning will be performed using the mass calibration compound FC43. Tuning will be performed to maximize the sensitivity of the mass spectrometer for the mass range of compounds being analyzed. In the FC43 spectra, the ion abundance of masses 131 and 219 are adjusted to a approximate ratio of 1:1. These two ions are then maximized to be approximately 50 to 70% of the ion abundance of the base mass 69. This procedure maximizes the sensitivity of the instrument in the mass region of interest for the PAH analysis.

10.3. A five-point initial calibration curve must be established showing the linear range of analysis. The same initial calibration is used for the two low level and the medium level ppt PAH analyses. The concentrations of standards used to construct the calibration curve are 20, 150, 300, 600, and 1200 ng/mL. The linear range for low level analysis (4 L to 0.5 mL) corresponds to sample concentrations of 2.5, 5, 30, 75, and 300 ng/L. If the concentration of any target compound in a sample exceeds the linear range defined by the above standards, the extracts must be diluted so that the concentrations of all target compounds fall within the range of the calibration curve. The linear range for medium level analysis (0.5 L to 5.0 mL) corresponds to final sample concentrations of 200, 400, 2400, 6000 and 12000 ng/L.

10.3.1. Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 12 hours.

10.4. If the initial calibration response factors are less than 35 relative percent difference sample analysis may proceed. If, for any analyte, the initial calibration response factor is greater than 35 relative percent difference the initial calibration curve must be repeated for that compound prior to the analysis of samples. The following compounds are excepted for this criteria due to poor response by this method: 7H-Dibenzo(c,g)carbazole, 3-Methylcholanthrene and the dibenzopyrene isomers.

10.5. Table II contains example RRT data for target compounds.

10.6. Continuing Calibration

Every 12 hours the mass spectrometer response for each PAH relative to the internal standard is determined using the 300 ng/mL calibration standard. The response factors for each compound must be compared to the initial calibration curve. If the continuing calibration response factors are within ± 35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the continuing calibration response factor is not within ± 35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples. The following compounds are excepted for this criteria due to poor response by this method: 7H-Dibenzo(c,g)carbazole, 3-Methylcholanthrene and the dibenzopyrene isomers.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Extraction

11.3.1. Samples are extracted at a pH >12.

11.3.2. For the low level extraction, a measured amount of sample, approximately 4 liters, is poured into either two 2-liter continuous liquid-liquid extractors, one 4-liter continuous liquid-liquid extractor, or two 4 liter separatory funnels. The surrogate solution is added, basified and the pH confirmed, and the samples are extracted with methylene chloride. The samples are shaken three times with 80 mL of methylene chloride for the shake-out technique. The samples are allowed to reflux for eighteen hours if the liquid-liquid extractor technique is used for preparation, with pH check. The extracts from each of the two-liter extractions for a sample are then combined for concentration.

11.3.3. The medium level extraction requires that 500 mL of the sample be extracted with methylene chloride for 18 hours in a one liter continuous liquid-liquid extractor or shaken three times with 60 mL of methylene chloride in a 2-liter separatory funnel.

11.3.4. The extracts are passed through an anhydrous sodium sulfate into a 500 mL Kuderna-Danish evaporative concentrator.

Note: The Kuderna-Danish glassware is to be rinsed with methylene chloride immediately prior to the addition of thc sample extract.

11.3.5. Concentrate the low level, ppt 75 and medium level extracts to approximately 5.0 mL using the Kuderna-Danish concentrator. Transfer the extracts to a calibrated N-Evap concentrator tube. The Kuderna-Danish tube is rinsed with methylene chloride. Transfer the rinsate to the N-Evap tube containing the sample extract.

11.3.6. Evaporate the extract using a nitrogen stream and a water bath at 30° to 35° C. Occasionally rinse the N-Evap tube walls with methylene chloride during this final concentration step. The low level and ppt 75 extracts are concentrated to 0.5 mL. The medium level extracts are concentrated to 5.0 mL.

11.3.7. Transfer the concentrated extracts to glass vials that are capped with a Teflon fitted septum. Store the extracts scparate from ppb level extracts.

11.4. Gas Chromatography/Mass Spectrometry Analysis

11.4.1. All aliquoting, extract dilutions, and spike additions must be performed in the trace laboratory using equipment dedicated to PAH-SIM analysis. Extract aliquots are added to 0.1 mL vials for GC/MS analysis to allow for re-analysis, if necessary.

11.4.2. Prior to analysis an aliquot of internal standard solution is transferred to the sample vial using a 25 µL syringe to give a final internal standard concentration of 600 ng/mL in the extract.

11.4.3. Representative aliquots are injected into the gas chromatograph/mass spectrometer using similar conditions to those provided in the following table. The injection technique should include 4 second hold time of the syringe needle in the injector after the sample has been injected.

Injector Temp	275°C
Transfer Line Temp	290°C
Initial Oven Temp	40°C
Initial Hold Time	0 min.
Ramp Rate	20°C/min. to 120°C; 17°C/min. to 150°C; 15°C/min. to 325°C – Hold for 8 min.
Final Temperature	325°C

11.4.4. The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each PAH as shown in Table I. All compounds detected at a concentration above the MDL are checked to insure the confirmation ion is present at the appropriate ratio.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

Obtain EICPs for the primary m/z and the confirmatory ion. The following criteria must be met to make a qualitative identification:

12.1.1. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

12.1.2. For the qualitative identification, the relative retention time (RRT) of unknown peaks must fall within ± 0.075 minutes.

12.1.3. The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. A compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive information includes correct relative retention time and the presents of the secondary ion but the ratio is greater than ± 20% of the primary ion which may be caused by an interference of the secondary ion. When the primary ion is not affected by interferences and the decision is agreed to by the reviewer, the compound is flagged with an asterisk (*) on the sample summary sheet.

12.1.4. Structural isomers that have very similar mass spectra and less than 30 second difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

12.2. Calculations

12.2.1. The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$$RF = \left(\frac{A_s \times C_{is}}{A_{is} \times C_s}\right)$$

Where:

A_s = Area of the characteristic ion for the parameter to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

C_{is} = Concentration of the internal standard, (ng/mL).

C_s = Concentration of the parameter to be measured, (ng/mL).

12.2.2. Based on these response factors, sample extract concentrations for each PAH is calculated using the following formula.

$$Ce = \left(\frac{A_s \times I_s}{A_{is} \times \overline{RF}}\right)$$

Where:

Ce = Sample extract concentration, ng/mL.

A_s = Area of the characteristic ion for the parameter to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

I_S = Amount of internal standard added to each extract, (ng/mL).

$\overline{RF}$ = The average response factor.

12.2.3. The actual sample concentration (C) for each compound is calculated by the following formula:

$$C = Ce \times \left(\frac{V_E}{V_s}\right)$$

Where:

C = Concentration of the sample, ng/mL.
V_E = The final extract volume, mL.
V_s = The original volume of sample extracted, L.
C_e = The amount measured in the analytical extract, ng/mL.

12.3. A second-level technical review of the organic data is performed prior to data reporting. This review is performed by a peer or supervisor using the guidelines and checklists detailed in SOP DEN-QA-0020.

13. METHOD PERFORMANCE

13.1. Method Detection Limit

A valid method detection limit (MDL) must be determined for each analyte of interest prior to analyzing samples and annually thereafter. MDLs are performed in laboratory reagent water or sand. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure must be segregated and disposed of according to the facility hazardous waste procedures (DEN-HS-0002). Waste will be disposed of in the appropriate satellite waste stream container. For this method, the following waste streams have been identified:

15.1. Expired Extract Vials – (Stream A)

15.2. Waste Dichloromethane – (Stream B)

15.3. Flammable Solvent – (Stream C)

15.4. Sodium Sulfate – (Stream D)

16. REFERENCES

16.1. SW846, Test Methods for Evaluating Soil Waste, Third Edition, Update III, December 1996, Method 3520C, and 8270C.

17. MISCELLANEOUS

17.1. A modification from the cited methods includc low level analysis which is subject to routine blank contamination with target constituents

17.2. Tables and Appendices:

Table I – Compounds and MS Quantitation Mass Ions

Table II – Example Relative Retention Times

Appendix I – Sample Preparation Flow Chart

Appendix II – Sample Analysis Flow Chart

Table I: Compounds and MS Quantitation Mass Ions*
Polynuclear Aromatic Hydrocarbons

Compound	Mass Ion	Confirmation Ion	Internal Standard Reference
Naphthalene	128	102	1
Acenaphthylene	152	151	1
Acenaphthene	154	153	1
Fluorene	166	165	1
Phenanthrene	178	176	2
Anthracene	178	176	2
Fluoranthene	202	200	2
Pyrene	202	200	2
Benzo(a)anthracene	228	226	3
Chrysene	228	226	3
Benzofluoranthenes	252	250	3
Benzo(a)pyrene	252	250	3
Indeno(1,2,3,cd)pyrene	276	138	3
Dibenz(a,h)anthracene	278	139	3
Benzo(g,h,i)perylene	276	138	3

Internal Standards

Compound	Mass Ion	Confirmation Ion	Internal Standard Reference
Acenaphthene-d_{10}	164	162	---
Phenanthrene-d_{10}	188	184	---
Benzo(a)pyrene-d_{12}	264	132	---

* The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum.

Table I: Compounds and MS Quantitation Mass Ions(Continued)
Surrogates

Compound	Mass Ion	Confirmation Ion	Internal Standard Reference
Naphthalene-d_8	136	134	1
Fluorene-d_{10}	176	174	1
Chrysene-d_{12}	240	236	3

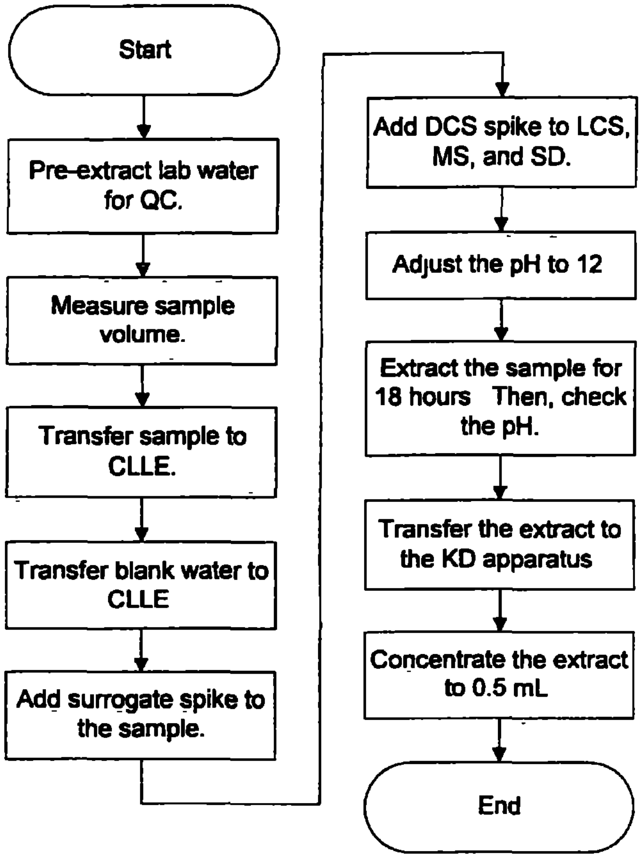
Heterocycles and Other PAH

Compound	Mass Ion	Confirmation Ion	Standard Reference
Indene	116	115	1
Indole	117	90	1
2,3-dihydroindene	117	118	1
2,3-benzofuran	118	90	1
Quinoline	129	102	1
Benzo(b)thiophene	134	89	1
2-methylnaphthalene	141	115	1
1-methylnaphthalene	141	115	1
Biphenyl	154	153	1
Carbazole	167	166	2
Dibenzofuran	168	139	1
Acridine	179	178	2
Dibenzothiophene	184	139	2
Perylene	252	250	3
Benzo(e)pyrene	252	250	3
7,12-Dimethylbenz(a)anthracene	256	241	3
3-Methylcholanthrene	268	252	3

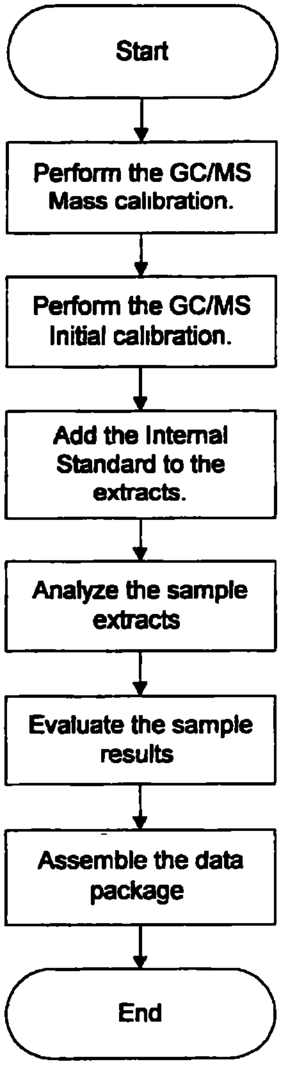
Table II: Example Relative Retention Times

Compound	Absolute Retention Time (minutes)
Benzofuran	4.64
Dihydroindene	5.04
Indene	5.14
Naphthalene-d_8(Surr.)	6.56
Naphthalene	6.58
Benzo(b)thiophene	6.66
Quinoline	7.15
Indole	7.69
2-methylnaphthalene	7.84
1-methylnaphthalene	8.02
Biphenyl	8.69
Acenaphthylene	9.47
Acenaphthene	9.79
Dibenzofuran	10.12
Fluorene-d_{10}(Surr.)	10.67
Fluorene	10.71
Dibenzothiophene	12.17
Phenanthrene	12.38
Anthracene	12.48
Acridine	12.54
Carbazole	12.75
Fluoranthene	14.42
Pyrene	14.80
Benz(a)anthracene	16.79
Chrysene-d_{12} (Surr.)	16.81
Chrysene	16.84
Benzofluoranthenes	18.46
Benzo(e)pyrene	18.86
Benzo(a)pyrene	18.93
Perylene	19.06
Indeno(1,2,3 cd)pyrene	20.82
Dibenz(ah)anthracene	20.86
Benzo(ghi)perylene	21.36

Appendix I: Sample Preparation Flow Chart



Appendix II: Sample Analysis Flow Chart



STL STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: CORP-MS-0001DEN, Revision 2.1)

Prepared by: Richard Burrows

Reviewed by: William Rhoades 8/29/00
Technical Specialist, William Rhoades

Approved by: Larry Penfold 8/29/00
Quality Assurance Manager, Larry Penfold

Approved by: Scott Kelly 8/30/00
Environmental Health and Safety Coordinator, Scott Kelly

Approved by: Timothy M. O'Shields 8/29/00
Laboratory Manager, Timothy M. O'Shields

Approved by: 8/29/00
Corporate Technology Scientist, Richard Burrows

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1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported ¾-methylphenol.
- Hexachlorophene and famphur analysis are not quantitative reliable by this method.
- Kepone should be analyzed by GC/ECD.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs Reporting limits will be proportionately higher for sample extracts that require dilution.

2 SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel. Solid samples are extracted with methylene chloride / acetone using sonication or soxhlet extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

Extraction procedures are detailed in SOP# CORP-OP-0001DEN. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3 DEFINITIONS

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the STL QC Program document (QA-003) for further details of the batch definition.

3.4 Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 LCS (Laboratory Control Sample) - A blank matrix (reagent water or Ottawa Sand) is spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method

3.8 Surrogates - Organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

4 INTERFERENCES

4.1 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause Method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

5.1 Procedures shall be carried out in a manner that protects the health and safety of all STL associates. The following requirements must be met:

5.1.1 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Gloves that have become contaminated will be removed and discarded. Disposable gloves shall not be reused.

5.1.2 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory The following specific hazards are known.

5.1.3 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

5.1.4 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.1.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported immediately to a laboratory supervisor.

6 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler. LEAP Technologies CTC A200S or equivalent.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used

6.6 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6 7 Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.8 Carrier gas: Ultra high purity helium.

7 REAGENTS AND STANDARDS

7.1 A minimum five point calibration curve is prepared when average response factors or linear regression curve fitting is used, six points for second-order fits. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal Standards are added to all standards and extracts to result in 40ng injected onto the column. For example, if the volume of an extract used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added for a 1 µL injection.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.

7.6 Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in 7.1 to 7.6 should be refrigerated at -10°C to -20°C if can be demonstrated that analytes do not fall out of solution at these temperatures. If not stable, the standards should be stored at 4 ± 2 °C. The standards must be replaced at least once a year. The continuing calibration standard should be replaced every week, when there are visible signs of degradation, or when the standard fails to meet Q.C. criteria. The continuing calibration standard is stored at ≤ 6°C.

8 SAMPLE PRESERVATION AND STORAGE

8.1 Reference appropriate facility SOP (DEN-QA-0003) for sample bottle, preservation, and storage requirements.

8.2 Samples are stored at 4 ± 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. Extracts are normally stored for 30 days after invoicing.

8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9 QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. MDL values used when reporting sample results are stored in the lab's LIMS.

9.1.2 For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. The minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration unless an alternative approach is agreed to with the client.

9.2 Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCS precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference + 3 standard deviations.

9.2.1 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is more than 4X.

9.2.2 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3 Refer to the QC program document (QA-003) for further details of control limits.

9.3 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001DEN for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except phthalate esters, as discussed in the next bullet) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client Alternatively, with advance client approval, the project reporting limit can be set at a higher level

- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.1 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.2 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.3 Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.3.4 Blank results are NOT subtracted from sample results.

9.4 Instrument Blank

9.4.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9 5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS is prepared using reagent water for aqueous methods and with Ottawa sand for solid sample methods. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 ng on-column depending on the analyte.

9.5 2 If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS). This type of justification should be reviewed and documented with the client before reporting.

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.3 Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted to concentrations below the calibration range.

9 7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. Surrogate compounds must be spiked at either 100 or 150 ng on-column, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2 If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

9.7.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate).

9.7.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the laboratory project manager and a facility QA representative.

9.9 Quality Assurance Summaries or Program Distillations

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries (also known as Program Distillations) should be developed to address these requirements.

9.10 STL QC Program

Further details of QC and corrective action guidelines are presented in the STL QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 CALIBRATION AND STANDARDIZATION

10.1 Summary

10 1.1 The instrument is tuned for DFTPP, calibrated initially with a five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1 Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. DDT breakdown must be < 20%. Refer to section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation

10.4.2 Compounds should be assigned to the IS with the closest retention time

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest when average response factors or linear regression curve fits are used. Six standards must be used for a quadratic least squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 40 ng on column. (For example, if the volume of the calibration standard used is 1 mL, add 100 µL of the 400 µg/mL internal standard solution for a 1 µL injection). The concentrations of all analytes are listed in tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCC's listed in 10.4.6.2) must be agreed with the client.

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the average of all RSDs in the initial calibration is $\leq$ 15%, then all analytes may use average response factor for calibration.

10.4.7.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.4.7.2 If the average of all the RSDs in the initial calibration is > 15%, then calibration on a curve must be used for those analytes with RSD > 15%. Linear or quadratic curve fits may be used. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

10.4.7.3 If a linear regression equation is used, the correlation coefficient r must be greater than 0.990. Use of second-order regression equations may be used on rare occasions, in which case the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination must be greater than 0.990.

10.4.8 Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.9 If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed Otherwise, proceed to continuing calibration.

10.4.10 Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.5 Calibration Verification

10.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 6.

10.5.2 Following a successful DFTPP analysis the continuing calibration verification standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration verification.

10.5.3 The following criteria must be met for the continuing calibration verification to be acceptable:

- The SPCC compounds must have a response factor of $\geq$ 0.05
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq$ 20%. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq$ 50%, with allowance being made for up to six target compounds to have percent drift greater than 50%.
- The internal standard response must be within 50-200% of the response in the level 3 of the most recent initial calibration sequence.
- If any internal standard retention time changes by more than 30 seconds from that of the level 3 of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrections made, as required.

10.5.3.1 If none of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCC's listed in 10.4.6.2) must be agreed with the client.

10.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11 PROCEDURE

11.1 Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification standard.

11.2.3 Add internal standard to the extract to result in 40 ng injected on column (for example, 50 µL internal standard solution in 0.5 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration verification.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see STL Policy # QA-011) or automatically by the data system. Minimum documentation required includes a hard copy of original data system peak integration and a similarly scaled hard copy showing the manual integration with analyst initials and date.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP # DEN-WC-0023 for determination of percent moisture.

11 7 Procedural Variations

11.7.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified before proceeding. The deviation shall be discussed in the final report case narrative. The Nonconformance Memo shall be filed in the project file Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.8 Troubleshooting Guide

11.8.1 Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the STL QAMP, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary
- Install new septum as necessary.

- Perform mass calibration as necessary.

11.8.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the ion volume or repeller, cleaning the source, replacing the multiplier and replacing the "top board" or RF-related electronics Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum

- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) and famphur

(CAS 52-85-7) fall into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1 Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. Estimated values are reported based on the response of the nearest internal standard.

12.2.2 Famphur

Display the mass chromatograms for mass 218 and mass 125 for the region of the chromatogram from two minutes before the fifth IS to 4 minutes after. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of famphur. Estimated values are reported based on the response of the nearest internal standard.

12.2.3 Methyl Chrysenes

Display the mass chromatogram for mass 242 for the region of the chromatogram from the 6th IS to the end of the run. Quantitation is based on the response of 6-methyl chrysene.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are.

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

- Molecular ions present in the reference spectrum should be present in the sample spectrum.

- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.

- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$\overline{RF}$ = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12 5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the RSDs of the response factors in the initial calibration is $\leq$ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.3.2 Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C= Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / \text{L} = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

W_S = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R=Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x=Area of the total ion chromatogram for the compound being measured

A_{is}=Area of the total ion chromatogram for the nearest internal standard without interference

RF=1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

13 METHOD PERFORMANCE

13.1 Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005. Method detection limit values used for reporting are stored in the lab's LIMS system.

13.2 Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

13.2.3 If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration should include the analysis of an extracted standard at the reporting limit and a single point calibration

13.4 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.5 Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14 POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15 WASTE MANAGEMENT

15.1 Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16 REFERENCES

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17 MISCELLANEOUS

17 1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17 1 3 This procedure includes the option for weighted linear regression curves using 1/concentration^2 weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on 1/standard deviation^2 weighting factors, which would require multiple analyses of each standard to determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance (standard deviation^2) is proportional to the standard concentration. EPA accepted this argument and indicated that they will issue a letter in July 1998, which will authorize the use of 1/concentration^2 weighting factors.

17.2 Modifications from Previous Revision

17.2.1 Section 9 3.4 is revised to clarify that blank subtraction is not allowed

17.2.2 Sections 9.1.1 and 13.1 are revised to explain where working MDL values are stored

17.2.3 Section 10.3.2 is rewritten to require the use of the DDT breakdown check in conjunction with every tuning event.

17.2.4 Section 10.4.7.3 has been added to include the linearity requirements when a regression equation is used to model the calibration.

17.2.5 Section 11.7.1 is revised to explain that any deviations from the SOP must be discussed in the final report case narrative.

17.2.6 Section 16.1 is revised to reference 8270C, rather than 8270B.

Tables

Table 1

STL Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330

Table 1

STL Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

[1] The STL primary standard is the standard normally used at STL. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients

[2] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2

STL Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3/4-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
Methyl Parathion[2]	298-00-0	50	1600

Table 2

STL Appendix IX[1] Standard Reporting Limits

4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion[2]	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin[3]	465-73-6	10	330
Famphur[3]	52-85-7	--	--
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

1 The Appendix IX standard contains additional analytes required for the Appendix IX list. The STL primary standard must also be analyzed to include all of the Appendix IX list.

2 May also be analyzed by method 8141, which can achieve lower reporting limits.

3 May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

Table 3

Reportable Analytes for STL Standard Tests, Primary Standard

Analyte	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	X
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X

Table 3

Reportable Analytes for STL Standard Tests, Primary Standard

4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[2]	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for STL Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2				X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
¾-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1				X
a,a-Dimethyl-phenethylamine	122-09-8				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin[2]	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp[2]	3689-24-5				X
Phorate[2]	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate[2]	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton[2]	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7				X
Methyl parathion[2]	298-00-0				X

Table 4

Reportable analytes for STL Standard Tests, Appendix IX Standard

4-Nitroquinoline-1-oxide	56-57-5				X
Parathion[2]	56-38-2				X
Isodrin[3]	465-73-6				X
Kepone [2,4]	143-50-0				X
Famphur [2,4]	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate[3]	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene[4]	70-30-4				X
Diphenylamine[5]	122-39-4				X

2 May also be analyzed by method 8141, which can achieve lower reporting limits

3 May also be analyzed by method 8081, which can achieve lower reporting limits

4 Hexachlorophene and famphur are analytes for Appendix IX. These compounds are not stable, and therefore not included in the calibration standard. The characteristic ions for these compounds are searched for in the chromatogram. (See section 12.2.1).

5 Diphenylamine is a required compound for Appendix IX N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished Diphenylamine is not included in the calibration standard.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	0.5 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3/4-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Parathion	109	97	291
Isodrin	193	66	195
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

[1] Levels are 50 and 75 ng/µL if 2 µL injection is used

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3/4-Methylphenol	100
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

[1] Levels are 50 ng/µL if 2 µL injection is used

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract[2]
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

[1] Included in standard mix, but not routinely evaluated for method 8270B

[2] Levels are 50 and 75 ng/µL if 2 µL injection is used

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Pyridine	--	20	50	80	120	160	200
N-nitrosodimethylamine	10	20	50	80	120	160	200
Aniline	10	20	50	80	120	160	200
Phenol	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	10	20	50	80	120	160	200
2-Chlorophenol	10	20	50	80	120	160	200
1,3-Dichlorobenzene	10	20	50	80	120	160	200
1,4-Dichlorobenzene	10	20	50	80	120	160	200
Benzyl alcohol	10	20	50	80	120	160	200
1,2-Dichlorobenzene	10	20	50	80	120	160	200
2-Methylphenol	10	20	50	80	120	160	200
2,2'-oxybis(1-chloropropane)[1]	10	20	50	80	120	160	200
4-Methylphenol	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	10	20	50	80	120	160	200
Hexachloroethane	10	20	50	80	120	160	200
Nitrobenzene	10	20	50	80	120	160	200
Isophorone	10	20	50	80	120	160	200
2-Nitrophenol	10	20	50	80	120	160	200
2,4-Dimethylphenol	10	20	50	80	120	160	200
Benzoic acid	--	--	50	80	120	160	200
Bis(2-chloroethoxy)methane	10	20	50	80	120	160	200
2,4-Dichlorophenol	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	10	20	50	80	120	160	200
Naphthalene	10	20	50	80	120	160	200
4-Chloroaniline	10	20	50	80	120	160	200
Hexachlorobutadiene	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	10	20	50	80	120	160	200
2-Methylnaphthalene	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	--	--	50	80	120	160	200
2,4,6-Trichlorophenol	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	10	20	50	80	120	160	200
2-Chloronaphthalene	10	20	50	80	120	160	200
2-Nitroaniline	--	--	50	80	120	160	200
Dimethyl phthalate	10	20	50	80	120	160	200
Acenaphthylene	10	20	50	80	120	160	200
3-Nitroaniline	--	--	50	80	120	160	200
Acenaphthene	10	20	50	80	120	160	200
2,4-Dinitrophenol	--	--	50	80	120	160	200
4-Nitrophenol	--	--	50	80	120	160	200
Dibenzofuran	10	20	50	80	120	160	200
2,4-Dinitrotoluene	10	20	50	80	120	160	200
2,6-Dinitrotoluene	10	20	50	80	120	160	200
Diethylphthalate	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	10	20	50	80	120	160	200

Table 12 (continued)
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Fluorene	10	20	50	80	120	160	200
4-Nitroaniline	--	--	50	80	120	160	200
4,6-Dinitro-2-methylphenol	--	--	50	80	120	160	200
N-Nitrosodiphenylamine	10	20	50	80	120	160	200
Azobenzene[2]	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	10	20	50	80	120	160	200
Hexachlorobenzene	10	20	50	80	120	160	200
Pentachlorophenol	--	--	50	80	120	160	200
Phenanthrene	10	20	50	80	120	160	200
Anthracene	10	20	50	80	120	160	200
Carbazole	10	20	50	80	120	160	200
Di-n-butyl phthalate	10	20	50	80	120	160	200
Fluoranthene	10	20	50	80	120	160	200
Benzidine	--	--	50	80	120	160	200
Pyrene	10	20	50	80	120	160	200
Butyl benzyl phthalate	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	--	--	50	80	120	160	200
Benzo(a)anthracene	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	10	20	50	80	120	160	200
Chrysene	10	20	50	80	120	160	200
Di-n-octylphthalate	10	20	50	80	120	160	200
Benzo(b)fluoranthene	10	20	50	80	120	160	200
Benzo(k)fluoranthene	10	20	50	80	120	160	200
Benzo(a)pyrene	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	10	20	50	80	120	160	200

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	20	50	100	160
N-Nitrosomethylethylamine	10	20	50	100	160
Methyl methanesulfonate	10	20	50	100	160
N-Nitrosodiethylamine	10	20	50	100	160
Ethyl methanesulfonate	10	20	50	100	160
Pentachloroethane	10	20	50	100	160
Acetophenone	10	20	50	100	160
N-Nitrosopyrrolidine	10	20	50	100	160
N-Nitrosomorpholine	10	20	50	100	160
o-Toluidine	10	20	50	100	160
3-Methylphenol	10	20	50	100	160
N-Nitrosopiperidine	10	20	50	100	160
o,o,o-Triethyl-Phosphorothioate	20	50	100	200	320
a,a-Dimethyl-phenethylamine	10	20	50	100	160
2,6-Dichlorophenol	10	20	50	100	160
Hexachloropropene	--	--	--	--	--
p-Phenylenediamine	10	20	50	100	160
n-Nitrosodi-n-butylamine	10	20	50	100	160
Safrole	10	20	50	100	160
1,2,4,5-Tetrachlorobenzene	10	20	50	100	160
Isosafrole 1 + 2	20	50	100	200	320
1,4-Dinitrobenzene	10	20	50	100	160
1,4-Naphthoquinone	10	20	50	100	160
1,3-Dinitrobenzene	10	20	50	100	160
Pentachlorobenzene	10	20	50	100	160
1-Naphthylamine	10	20	50	100	160
2-Naphthylamine	10	20	50	100	160
2,3,4,6-Tetrachlorophenol	10	20	50	100	160
5-Nitro-o-toluidine	10	20	50	100	160
Thionazin	10	20	50	100	160
1,3,5-Trinitrobenzene	20	50	100	200	320
Sulfotepp	10	20	50	100	160
Phorate	10	20	50	100	160
Phenacetin	10	20	50	100	160
Diallate 1 + 2	20	50	100	200	320
Dimethoate	10	20	50	100	160
4-Aminobiphenyl	10	20	50	100	160
Pentachloronitrobenzene	20	50	100	200	320
Pronamide	10	20	50	100	160
Disulfoton	10	20	50	100	160
2-secbutyl-4,6-dinitrophenol (Dinoseb)	20	50	100	200	320
Methyl parathion	10	20	50	100	160
4-Nitroquinoline-1-oxide	20	40	100	200	320
Parathion	10	20	50	100	160

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Isodrin	10	20	50	100	160
Famphur	--	--	--	--	--
Methapyrilene	10	20	50	100	160
Aramite 1 and 2	20	50	100	200	320
p-(Dimethylamino)azobenzene	10	20	50	100	160
p-Chlorobenzilate	10	20	50	100	160
3,3'-Dimethylbenzidine	10	20	50	100	160
2-Acetylaminofluorene	10	20	50	100	160
Dibenz (a,j)acridine	10	20	50	100	160
7,12-Dimethylbenz(a)anthracene	10	20	50	100	160
3-Methylcholanthrene	10	20	50	100	160

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100 0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68 1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71 6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70 9-109.4
Hexachlorobenzene	60	24 9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46 6-180.2
Naphthalene	60	30.1	35.6-119 6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55 4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28 7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106 5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129 2

[1]Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270C, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of

capability for method 8270B.

APPENDIX A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

18 REQUIREMENTS FOR METHOD 625

18.1 Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits or other CWA compliance situations. The standard analytc list and reporting limits are listed in Table A-1.

18.2 This method can be applied only to aqueous matrices.

18.3 The tune period for this method is defined as 24 hours

18.4 Initial calibration curve requirements:

18.4.1 The initial calibration curve for this method requires at least three points.

18.4.2 Target compounds must have RSD ≤ 35%.

18.4.3 If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

18.5 Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

18.6 Matrix Spike and LCS requirements.

18.6.1 A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1. STL Method 625 standard reporting list and reporting limits.

Analytes	CAS Number	Aqueous µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10

Analytes	CAS Number	Aqueous µg/L
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2. Method 625 LCS and MS compounds and spike concentrations.

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100

LCS Compounds	Spiking Level, ng/µL in extract[1]
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrenc	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

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Implementation Date ______________

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: SAMPLE MANAGEMENT AND CHAIN-OF-CUSTODY

(SUPERSEDES: REVISION 6.0)

Prepared by: Larry Penfold

Reviewed by: 9-28-00
Technical Specialist, Jeff Mott

Approved by: 9-28-00
Quality Assurance Manager, Larry Penfold

Approved by: 9/29/00
Environmental Health and Safety Coordinator, Scott Kelly

Approved by: 9/29/00
Laboratory Director, Timothy M. O'Shields

1. PURPOSE AND SCOPE

This procedure describes the management of samples throughout the laboratory, from receipt of samples through archiving of old samples. Included are verification of sample identity, chain-of-custody documentation, verification of sample condition and preservation, notification to the lab of short-holding-time samples, resolution of sample receipt discrepancies, requirements for sample delivery acceptance, and sample archiving.

Two receiving areas exist in the laboratory, one for samples without significant levels of radioactivity and one for radioactive samples. The locations are indicated on the facility map in Figure 8.11-1 in the Laboratory Quality Manual. The outside of the building is marked to make the distinction for people delivering samples. Clients are instructed by the laboratory Project Manager which of the two areas will take delivery of their samples. Additional training and procedures, as specified in the Radiation Safety Manual, are required for personnel receiving radioactive samples. Details for screening, monitoring and controlling radioactive samples are described in SOP# AU-HPM-R001, "Standard Operating Procedures for Radioactive Sample Control and Screening." SOP# AU-HPM-R001 is complimentary with this SOP, meaning that all of the processes described in this SOP apply to both sample receiving areas.

2. RESPONSIBILITIES

Sample Receiving Personnel - They are responsible for signing the Chain-of-Custody forms upon receipt of samples, completing the Sample Checklist, noting short-holding-time parameters when indicated on paperwork from the client, documenting discrepancies on the Condition Upon Receipt Anomaly (CUR) form, promptly notifying Project Managers of all discrepancies, and maintaining the samples in the walk-in cooler and sample archive area.

Revised 4/01/17/01
4/01/17/01

All personnel are required to take basic radiation training. Any coolers or samples that have radiation stickers or paperwork indicating possibly significant levels of radioactivity are required to move the samples to the Mixed Waste Sample Receiving area.

2.2. Project Manager – The Project Manager (PM) must build a quote in the QuantIMS database before samples can be logged into the system. The quote includes information about the types of samples expected, extraction/digestion methods, and analytical methods to be used. The PM provides Sample Receiving staff with the quote number to use as the basis for logging in samples. The PM contacts the client to resolve all sample receipt discrepancies, documents on the CUR any decisions reached, and instructs Sample Receiving staff accordingly.

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Implementation Date 01/17/01

STL-Denver

Interim SOP Change - This is a controlled document

SOP NUMBER: **DEN-QA-0003**

SOP TITLE: Sample Management and Chain-of-Custody
SOP SECTION(S) AFFECTED BY CHANGE: Section 2
REASON FOR CHANGE(S): Correct numbering
Change section ~~(Attachment F)~~ 2 y01/17/01 **from:** As it currently reads **To:** Number sections as indicated 2.1 Sample receiving personnel..... 2.2 All personnel are required to take...... 2.3 Project Manager......
SUBMITTED BY/DATE: Robert Hanisch 01-08-01
APPROVED BY:
Robert CHanisch **Technical Reviewer** **Date** 1/8/01
James S. Kelly, Health and Safety Coordinator **Date** 1/8/01
Larry Penfold, Quality Assurance Manager **Date** 1/17/01
Timothy M. O'Shields, Laboratory Director **Date** 1/17/01

3. SAFETY

3.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

3.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

3.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory

3.4. Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

3.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

Do not open any shipping containers marked "Biohazard" or "Extremely Hazardous" before consulting management personnel.

Segregate High Level / High Hazard Samples - The following types of incoming samples should be segregated in refrigerators separate from other samples to avoid contamination. In addition, the Health & Safety Officer should be called in to evaluate the samples and the need for additional safety precautions.

- Samples containing visible amounts of non-aqueous liquid (e.g. solvents, petroleum products, etc.)
- Samples labeled with a radiation sticker
- Samples labeled with a biohazard sticker
- Samples marked as "high hazard" or "extremely hazardous", or other phrases that indicate that special safety problems may exist.

4. PROCEDURE

4.1. Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

4.2. Sample Receipt

4.2.1. Sample shipments arrive at the central shipping and receiving area. The delivery person rings the exterior bell to have one of the Sample Receiving staff unlock the door.

4.2.2. Sample Receiving signs the shipping receipt or freight bill to indicate receipt of the coolers. The freight bill is an important part of the chain-of-custody record for the samples, and whenever possible must be kept as part of the permanent project records.

4.2.4. For the protection of the people working in the area, it is important to first open the coolers in the walk-in hood. Once it has been determined that there are not broken samples and no fumes coming from the cooler the cooler can be moved to the sink or to one of the benches.

4.2.5. Use the Sample Receiving Checklist (Attachment A) to record the following initial checks:

4.2.5.1. Client name and sampling site.

4.2.5.3. Condition of cooler custody seals, if present.

4.2.5.4. Presence or absence of custody form(s).

4.2.5.5. Chain-of-custody (COC), Attachment B, has necessary dates and signatures for earlier sample transfers.

4.2.5.6. Temperature inside coolers

4.2.5.6.1. Two types of temperature measuring devices are used in the area. Both types are calibrated daily (see calibration procedures in SOP # DEN-QA-0001). The infrared (IR) temperature gun is used to take an instantaneous temperature reading from the surface of the container. The IR gun should be 1-3" from the sample when the reading is taken. The thermocouple temperature probes are used to take a reading after equilibrating approximately 2 minutes or until the temperature reading has stabilized.

4.2.5.6.2. If the cooler contains a temperature blank, take a reading from that bottle.

- If a thermocouple probe is used, it should be placed directly into the water in the bottle. The IR gun can be used to take an instantaneous reading without opening the bottle.
- If the cooler does not contain a temperature blank and a thermocouple probe is used, place the probe next to a sample bottle and take a reading after 2 minutes or after the temperature reading has stabilized.

4.2.5.6.4. Record the temperature of each cooler on the Sample Receiving Checklist. If the temperature in the cooler is greater than 6° C, record this observation as a deviation in the comments section of the Sample Checklist sheet, and prepare a Condition Upon Receipt Anomaly Report (CUR).

4.2.6. Unpack the cooler, and line up the bottles in the numerical order given on the COC.

4.2.7. Photograph all broken sample containers and multiphase samples.

4.2.8. Empty the cooler of all packing materials. Carefully check for additional samples or documents before discarding the packing.

4.2.9. Check and record the following information on the Sample Receiving Checklist:

4.2.9.1. Labels are present and intact.

4.2.9.2. pH of samples meets requirements:

4.2.9.2.1. Refer to "Guidelines for Sample Bottles and Preservatives" (Attachment D) to determine what the pH should be.

4.2.9.2.2. Do not perform pH check on samples marked for volatile organic tests. These will be done later by the VOA analysts.

4.2.9.2.3. For non-volatile tests requiring chemical preservation, open the bottle, remove a few tenths of a milliliter of sample with a disposable transfer pipette, touch the pipette tip to wide-range (0-14) pH strips. Compare the color that develops immediately to the color chart.

4.2.9.2.4. Discard the pipette and any unused sample.

4.2.9.2.5. Initial the form indicating that the pH check was performed on all samples.

4.2.9.2.6. List the sample number and pH for any samples not meeting requirements.

4.2.9.3. Number of containers indicated on custody form matches the number received.

4.2.9.4. Client sample identifiers on the container labels exactly matches identifiers on the custody form.

4.2.9.5. Volatile organic vials are completely filled, no bubbles.

4.2.9.6. Proper preservation marked on VOA vials.

4.2.9.7. Presence of sediment in samples that the COC indicates was filtered in the field.

4.2.9.8. Client supplied extra volume for QC on at least one sample.

4.2.9.9 Presence of multiple phases in any of the samples (e.g., solids and liquid or multiple liquid phases).

4.2.10. Verify that the sample volumes meet requirements.

4.2.11. Corrective action for discrepancies

4.2.11.1. Any discrepancies must be noted on a Condition Upon Receipt Anomaly Report (see Attachment C).

4.2.11.2. The PM must be notified as soon as possible, normally the day received, so that the client may be contacted in a timely manner.

4.2.11.3. Completion of the CUR report by the PM is part of the log-in process, which is not complete until this is done.

4.3. Sample Login

4.3.1. Sign on to the QuantIMS terminal.

4.3.2. Select the login option for new samples under the client and program indicated on the PM's quotation for the work.

4.3.3. The program will automatically assign the next lot number.

4.3.4. Label the COC with the lot number, and assign sequential sample numbers beginning with the number one.

4.3.5. Print labels and attach them to the sample bottles.

Revised 4/01/12/01

4.3.6. A second person must verify that sample labels, both the client's sample numbers and STL's, on the containers exactly match the COC.

4.3.7. Enter all the client and sample information requested by the log-in program. Assign analysis codes based on the quote prepared by the PM.

4.3.8. If the project includes any tests with short holding times, fill out a Short Holding Time Form (Attachment E), and immediately delivered to the work area. A signature from the area accepting the samples is required before work can proceed.

4.3.9. Print a Client Analysis Summary from QuantIMS.

4.3.10. A folder is created for the sample lot. A complete folder contains:

- A manila file folder labeled with the lot number
- Chain-of-custody forms
- Freight bills
- The completed Sample Checklist
- A CUR report, if used
- A photograph for any broken bottles or multiphase samples
- Short Hold Forms, if used
- Client Analysis Summary, which lists the information entered into QuantIMS for each sample
- Any paperwork the client supplied with the samples

4.3.11. Deliver the project file to the PM for review and approval.

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Implementation Date 01/17/01

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Interim SOP Change - This is a controlled document

SOP NUMBER: **DEN-QA-0003**

SOP TITLE: Sample Management and Chain-of-Custody
SOP SECTION(S) AFFECTED BY CHANGE: 4.3.5
REASON FOR CHANGE(S): To properly preserve VOA water samples.
Change section *(4.3.5)* **from:** As it reads **To:** Add the following: **NOTE:** If it is suspected that the temperature of the VOA vials will be compromised, i.e. out on the counter more than approximately 30 minutes, place the VOA samples in the sample receiving VOA refrigerator until samples are ready to be labeled and place in the appropriate refrigerator.
SUBMITTED BY/DATE: V. Rhonda Johnson 01/16/01
APPROVED BY:

Technical Reviewer	**Date** 1-17-01
James S. Kelly, Health and Safety Coordinator	**Date** 1/17/01
Larry Penfold, Quality Assurance Manager	**Date** 1-17-01
Timothy M. O'Shields, Laboratory Director	**Date** 1/17/01

4.3.12. The PM works with the client and Sample Receiving, as needed, to resolve any uncertainties in log-in information.

4.4. Sample Storage

4.4.1. Samples are boxed by type (i.e., according to test group).

4.4.2. Samples are stored in the walk-in cooler, except for

- Samples for volatile organics analysis (VOA), which are taken directly to the VOA area refrigerators,
- Water samples for metals analysis, which go directly to the metals group,
- Samples for projects that require storage in the locking refrigerators.

4.4.3. The shelf number where samples are stored in the walk-in cooler is written on the sample labels, on the box of samples, and on the Internal Chain-of-Custody form.

4.4.4. Consult the Quarantine Sample SOP for proper storage of samples or coolers labeled with "Quarantine Sample" stickers or other USDA labels. A special set of shelves in the walk-in cooler is marked for storing quarantine samples.

Subcontract Work

4.5.1 STL has a list of approved laboratories that may be used for testing not performed by this lab (the approval process is described in SOP # CORP-QA-0012 "Selection and Evaluation of Subcontractor Laboratories").

4.5.2 The PM indicates any subcontract lab arrangements in the quotation.

4.5.3 Separate COCs must accompany the samples sent out to other labs. This COC must clearly indicate sample identities and the tests that must be performed. Shipping personnel sign the COC to document relinquishing the samples to the freight company.

4.5.4 When the samples have been shipped, a copy of the COC is forwarded to the PM.

4.6 Internal Chain of Custody

4.6.1. The Internal Chain-of-Custody (ICOC) form (Attachment E) is used to document the movement of samples from central storage to other areas in the laboratory. Instructions for completing the ICOC form are shown in Attachment F. The forms are kept in binders at the entrance to the Sample Receiving area. Analysts document the receipt of samples as they are removed from the walk-in cooler, and as the samples are returned to the cooler. Samples should be returned to the cooler soon after sample aliquots have been taken. All samples should be returned to the cooler by the end of the day.

4.6.2. There are three satellite sample storage areas in the lab, metals, MS-VOAs and GC-VOAs. Whenever samples are stored in these areas, their transfer must be recorded on the ICOC and a copy of the ICOC must accompany the samples to the satellite storage area. Each group with a satellite sample storage area is responsible for maintaining the ICOC forms in the area and properly documenting when the samples are archived. Under standard ICOC procedures it is not required to record each time a sample in a satellite area is handled. This is because the samples stored in the satellite area are assigned directly to the applicable group and are to be used only by that group, thus the samples are assumed to be in the custody of the responsible group at all times. This is in contrast to the samples stored in the walk-in which are not in the direct custody of one group, thus it is required that each time a sample stored in the walk-in is handled it must be recorded on the ICOC.

4.6.3. For some projects, maximum security of samples beyond storage in the secured building or a satellite area is required. This is called strict internal chain-of-custody. Under strict internal chain-of-custody procedures, the analyst name and the date and time the sample was removed from and returned to storage must be documented on the Internal Chain-of-Custody form, regardless of the area the sample is stored. When strict internal chain-of-custody is required, the sample custodian must check the "Strict Internal COC Required" area on the bottom of the ICOC form prior to distributing the ICOC to the satellite areas and placing it outside of the sample receiving area.

4.6.3.1. The project managers must inform the sample receiving group of all clients who require strict internal COC and include such information on the QuantIMS checklists.

4.6.3.2. Upon receipt at the satellite areas, it is the responsibility of the analysts in the group to follow the Strict ICOC procedures. The completed form(s) becomes part of the project file.

4.6.4. Samples are kept in the walk-in cooler and the satellite areas until final results have been reported to the client. After which, the samples are moved to the sample archive area. This transfer of samples is also recorded on the ICOC form.

4.7 Sample Archiving

4.7.1 An entry is made in the Aqueous/Soil Sample Disposal Log as samples are moved into and out of the sample archive area. Entries include project or lot number, storage shelf number, date samples placed in archive storage, disposal date, and initials.

4.7.2 Solid samples scheduled for disposal are categorized as "Non-Regulated Soil Waste" (see SOP # DEN-CHP-001 "Waste Characterization and Categorization Procedure" for details), and are placed in 55 gallon drums in the hazardous waste storage area. These wastes are disposed of by destructive incineration. Each day solids are out of the archive area, an entry is made in the "Sample Drum Inventory Log" to record what drum is receiving solids for the day.

4.7.3 Further details concerning waste management procedures and personnel training requirements are described in SOP # DEN-CHP-0002 "Waste Collection, Accumulation and Storage Procedure" and SOP # DEN-CHP-003 "Waste Shipping and Manifesting Procedure." All work performed in the hazardous waste storage area must be done under the direction of the Environmental Health and Safety Officer.

5. DEFINITIONS

5.1. Chain-of-Custody Form: A critical legal document, which records collection, possession, and transfer of samples from one person or party to another.

Sample Delivery Acceptance: The point in time at which STL is first obligated to initiate preparation and/or analysis of samples.

Internal Chain-of-Custody Form: The form used to document the storage, handling and archival of client samples while in possession of the STL-Denver laboratory.

Strict internal chain-of-custody: Requirement that each time a sample is handled within the laboratory, the analyst name and date and time the sample was removed from and returned to storage must be documented on the Internal Chain-of-Custody form, regardless of the area the sample is stored. This requirement is in addition to standard internal chain-of-custody requirements and is only implemented by a specific client request or program requirement.

MISCELLANEOUS

Changes From the Previous Version of the SOP

The Purpose and Scope section is revised to include references to other documents describing the area, training, and handling that apply to radioactive samples.

6.1.1 Units (°C) added to cooler temperature line on checklist.

6.1.2 N/A box added to checklist for pH of samples (the check is only done for water samples).

6.1.3 A line was added to the checklist for the Project Manager to indicate if water samples require a residual chlorine check (samples from a potentially chlorinated source).

6.2. Attachments

Attachment A:	Sample Receiving Checklist
Attachment B:	STL Chain of Custody
Attachment C:	Condition Upon Receipt Anomaly Form
Attachment D:	Guidelines for Sample Bottles and Preservatives
Attachment E:	Short Holding Time Form
Attachment F:	Internal Chain-of-Custody Form
Attachment G:	Instructions for the Use of the Internal Chain-of-Custody Form
Attachment H:	Flow Chart

ATTACHMENT A

SAMPLE RECEIVING CHECKLIST

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Interim SOP Change - This is a controlled document

SOP NUMBER: **DEN-QA-0003**

SOP TITLE: Sample Management and Chain-of Custody
SOP SECTION(S) AFFECTED BY CHANGE: Attachment A (Sample Receiving checklist)
REASON FOR CHANGE(S): Update current practices.
Change section *[insert section #]* **from:** As is **To:** Addition of item #21
SUBMITTED BY/DATE: 11/3/00
APPROVED BY:
[signature] Technical Reviewer — 11-3-00 Date
[signature] James S. Kelly, Health and Safety Coordinator — 11/13/00 Date
[signature] Larry Penfold, Quality Assurance Manager — 11/3/00 Date
[signature] Timothy M. O'Shields, Laboratory Manager — 11/13/00 Date

STL Denver

Sample Receiving Checklist

Lot #:______________________ Date/Time Received:______________________

Company Name & Sampling Site:______________________

*Cooler #(s): ________ ________ ________ ________ ________ ________ ________ ________

Temperatures (°C): ________ ________ ________ ________ ________ ________ ________ ________

PM to Complete This Section: *Yes* *No*

Residual chlorine check required: ☐ ☐

Special instruction to Sample Receiving staff:

Unpacking & Labeling Check Points:

N/A	*Yes*	*No*		*Initials*
	☐	☐	1. Cooler seals intact.	________
	☐	☐	2. Chain of custody present.	________
	☐	☐	3. Bottles broken and/or are leaking, comment if yes.	________
			PHOTOGRAPH BROKEN BOTTLES	
	☐	☐	4. Containers labeled, comment if no.	________
☐	☐	☐	5. pH of all samples checked and meet requirements, note exceptions.	________
	☐	☐	6. Chain of custody includes "received by" and "relinquished" by signatures, dates, and times.	________
	☐	☐	7. Receipt date(s) > 48 hours past the collection date(s)? If yes, notify PA/PM.	________
	☐	☐	8. Chain of custody agrees with bottle count, comment if no.	________
	☐	☐	9. Chain of custody agrees with labels, comment if no.	________
☐	☐	☐	10. VOA samples filled completely, comment if no.	________
☐	☐	☐	11. VOA bottles preserved, check for labels.	________
	☐	☐	12. Did samples require preservation with sodium thiosulfate?	________
☐	☐	☐	13. If yes to #12, did the samples contain residual chlorine?	________
☐	☐	☐	14. Sediment present in "D," dissolved, bottles.	________
	☐	☐	15. Are analyses with short holding times requested?	________
☐	☐	☐	16. Is extra sample volume provided for MS, MSD or matrix duplicates?	________
	☐	☐	17. Multiphase samples present? If yes, comment below.	________
	☐	☐	18. Any subsampling for volatiles? If yes, list samples.	________
			PHOTOGRAPH MULTIPHASE SAMPLES	
☐	☐	☐	19. Subcontract COC signed and sent with samples to bottle prep?	________
	☐	☐	20. Was sample labeling double checked by a second person?	________

Revised 11/13/00

Document ~~any problems or~~ discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR).

STL Denver

Sample Receiving Checklist

Lot #:______________________ Date/Time Received:______________________

Company Name & Sampling Site:______________________

*Cooler #(s): ______ ______ ______ ______ ______ ______ ______ ______

Temperatures (°C): ______ ______ ______ ______ ______ ______ ______ ______

PM to Complete This Section: *Yes* *No*

Residual chlorine check required: ❑ ❑

Special instruction to Sample Receiving staff:

Unpacking & Labeling Check Points:

N/A	*Yes*	*No*		*Initials*
	❑	❑	1. Cooler seals intact.	______
	❑	❑	2. Chain of custody present.	______
	❑	❑	3. Bottles broken and/or are leaking, comment if yes.	______
			PHOTOGRAPH BROKEN BOTTLES	
	❑	❑	4. Containers labeled, comment if no.	______
❑	❑	❑	5. pH of all samples checked and meet requirements, note exceptions.	______
	❑	❑	6. Chain of custody includes "received by" and "relinquished" by signatures, dates, and times.	______
	❑	❑	7. Receipt date(s) > 48 hours past the collection date(s)? If yes, notify PA/PM.	______
	❑	❑	8. Chain of custody agrees with bottle count, comment if no.	______
	❑	❑	9. Chain of custody agrees with labels, comment if no.	______
❑	❑	❑	10. VOA samples filled completely, comment if no.	______
❑	❑	❑	11. VOA bottles preserved, check for labels.	______
	❑	❑	12. Did samples require preservation with sodium thiosulfate?	______
❑	❑	❑	13. If yes to #12, did the samples contain residual chlorine?	______
❑	❑	❑	14. Sediment present in "D," dissolved, bottles.	______
	❑	❑	15. Are analyses with short holding times requested?	______
❑	❑	❑	16. Is extra sample volume provided for MS, MSD or matrix duplicates?	______
	❑	❑	17. Multiphase samples present? If yes, comment below.	______
	❑	❑	18. Any subsampling for volatiles? If yes, list samples.	______
			PHOTOGRAPH MULTIPHASE SAMPLES	
❑	❑	❑	19. Subcontract COC signed and sent with samples to bottle prep?	______
	❑	❑	20. Was sample labeling double checked by a second person?	______
❑	❑	❑	21. Were sample bottles, COC and other paperwork double checked for dissolved metals by a second person?	______

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR).

ATTACHMENT B

STL CHAIN-OF-CUSTODY FORM

Chain of Custody Record

SEVERN TRENT SERVICES

Severn Trent Laboratories, Inc.

STL-4124 (0700)

Client	Project Manager	Date	Chain of Custody Number 017145
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page ______ of ______

City	State	Zip Code	Site Contact	Lab Contact	Analysis (Attach list if more space is needed)
Project Name and Location (State)			Carrier/Waybill Number		
Contract/Purchase Order/Quote No			Matrix	Containers & Preservatives	

Special Instructions/ Conditions of Receipt

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Air	Aqueous	Sed.	Soil		Unpres.	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/ NaOH													Special Instructions/ Conditions of Receipt

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For ______ Months (A fee may be assessed if samples are retained longer than 3 months)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other ______

QC Requirements (Specify)

	Date	Time		Date	Time
1. Relinquished By			1. Received By		
2. Relinquished By			2. Received By		
3. Relinquished By			3. Received By		

Comments

DISTRIBUTIO[N: W]HITE - Stays with the Sample; CANARY - Returned to Client with Report; PINK - Field Copy

ATTACHMENT C

CONDITION UPON RECEIPT ANOMALY FORM

STL Denver
Condition Upon Receipt Anomaly Report (CUR)

Client: ____________________ Date/Time: ____________________

Lot No: ____________________ Initiated by: ____________________

Affected Samples COC# ____________

Client ID	Lab ID	Analyses Requested

CONDITION/ANOMALY/VARIANCE (CHECK ALL THAT APPLY):

❑COOLERS
- ❑Not Received, No Chain of Custody (COC)
- ❑Not Received but COC(s) Available
- ❑Leaking
- ❑Other: ____________

❑CUSTODY SEALS (COOLER(S)/CONTAINER(S)
- ❑None
- ❑ Not Intact
- ❑Other: ____________

❑ TEMPERATURE (greater than 6° C)
- ❑Cooler Temp ____________
- ❑Temperature Blank ____________

❑CHAIN OF CUSTODY (COCs)
- ❑Not relinquished by Client; No date/time Relinq.
- ❑ Incomplete Information
- ❑Other: ____________

❑CONTAINERS
- ❑Leaking
- ❑Broken
- ❑Extra
- ❑Without Labels
- ❑VOA Vials with Headspace ____________mm
- ❑Other: ____________

❑ CONTAINER LABELS
- ❑Not the same ID/info as in COC
- ❑ Incomplete
- ❑ID COLLECTION ❑Time ❑Date ❑PRESERVATIVE
- ❑ Markings/Info smeared or illegible
- ❑Torn
- ❑Other: ____________

❑ SAMPLES
- ❑Samples NOT RECEIVED but listed on COC ------ ❑will be noted on COC ❑ Client to send samples with new COC
- ❑Samples received but NOT LISTED on COC
- ❑Logged based on Label Information
- ❑Logged based on info from other samples on COC
- ❑ Logged according to Work Plan
- ❑Logged on HOLD UNTIL FURTHER NOTICE
- ❑ Other ____________
- ❑ Mislabeled as to tests, preservatives, etc.
- ❑Holding time expired
- ❑ Improper container used
- ❑Not preserved / Improper preservative used
- ❑ Improper pH ________ ❑ Lab to preserve sample
- ❑ Insufficient quantities for analysis

Comments: ____________________

Corrective Action:
- ❑ Client Informed: verbally on: ________By: ________: In writing on: ________ By: ________
- ❑ Sample(s) processed "as is". ____________
- ❑ Sample(s) on hold until: ____________ If released, notify: ____________

Sample Control Supervisor Review: ____________________ Date: ____________

Project Management Review: ____________________ Date: ____________

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

ATTACHMENT D

GUIDELINES FOR SAMPLE BOTTLES AND PRESERVATIVES

SAMPLE BOTTLES AND PRESERVATIVES

(Unless otherwise noted all samples must be cooled to 4 °Celsius)

References 40 CFR 136 3, Table II

Aqueous Matrices

	Parameter	Container	Chemical Preservatives
1	Alkalinity, BOD, Chloride, Color, Residual Chlorine, Chromium VI, Conductance, Fluorine, MBAS, Nitrite, Orthophosphate, Total Dissolved/Suspended Solids, Sulfate, Sulfite, pH, Nitrate	1000 mL poly HDPE (NM)	None
2	Ammonia, COD, TKN, TON, Nitrate + Nitrite, Total Phosphate, TOC, Phenolics	500 mL glass (BR)	2 mL 50% Sulfuric Acid, pH <2
3	TPH, Oil & Grease	Two (2) 1000 mL glass (BR)	4 mL 50% Sulfuric Acid, pH <2
4	Metals (excluding Hg), Hardness	500 mL HDPE (NM)	10 mL 20% Nitric Acid, pH <2
4 C	CLP Metals (excluding Hg), Hardness	1000 mL HDPE (NM)	20 mL 20% Nitric Acid , pH <2
5	Gross Alpha, Gross Beta, ^3H	Two (2) 1000 mL HDPE (NM)	20 mL 20% Nitric Acid, pH <2
6	Total and/or Free Cyanide	250 mL HDPE (NM)	2 mL 50% Sodium Hydroxide, pH > 12
6 C	CLP Total and/or Free Cyanide	500 mL HDPE (NM)	4 mL 50% Sodium Hydroxide, pH >12
7	Sulfide	Two (2) 250 mL HDPE (NM)	1 mL 1 N Zinc Acetate **and** 1 mL 50% Sodium Hydroxide, pH > 9
9	Volatile Hydrocarbons, GRO	Three (3) 40 mL glass	200 µL Hydrochloric Acid, pH < 2
11	Purgeable Organics	Three (3) 40 mL glass	200 µL HCl (pH < 2), if sample is chlorinated add 100 µL 2% Sodium Thiosulfate
12	Base, Neutral, Acid Compounds, Dioxins	Two (2) 1000 mL amber glass (BR)	None
13	Pesticides, PCBs	Two (2) 1000 mL glass (BR)	None
14	Herbicides	Two (2) 1000 mL glass (BR)	None
15	TOX	One (1) 1000 mL amber glass	4 mL 50% Sulfuric Acid (pH < 2), if sample is chlorinated add 100 µL 2% Sodium Thiosulfate
16	Extractable Hydrocarbons, DRO	Two (2) 1000 mL glass (BR)	None
20, 21	Bulk Water Analysis	1/2 gallon or 1 gallon glass (WM)	None

Solid Matrices

30	Organics, TPH, Metals, Radiochemistry, Oil & Grease	500 mL glass (WM)	None
31	Wet Chemistry, not listed for 30	250 mL glass (WM)	None
32	Purgeable Organics	125 mL glass (WM)	None

TCLP/SLP Matrices

33	Purgeable Organics (Solid phase)	Two (2) 125 mL glass (WM)	None
	All other analytes (Solid phase)	1000 mL amber glass (WM)	None
34	Purgeable Organics (Multiphase)	Two (2) 250 mL glass (WM)	None
	All other analytes (Multiphase)	Four (4) 1000 mL amber glass (WM)	None

NM - Narrow Mouth, BR - Boston Round, WM - Wide Mouth, HDPE - High Density Polyethylene, ⊗ - Does not require temperature preservation

ATTACHMENT E

SHORT HOLDING TIME FORM

STL Denver

Short Holding Time Form

Lot Number: ________________ Client: ____________________.

Internal Tracking Record (to be completed be each person handling the samples)

	Initials	Date	Time	Comments
Sample Receiving				
Project Manager				
Wet Chem. Rep.				
Analyst				

Holding Time	Analysis	Method No.	Sample Number(s)	
Analyze Immediately On Collection (testing for these parameters should be done in the field)	Dissolved Oxygen	360.1		
	Free Carbon Dioxide (CO2)	4500-CO2		
	Sulfite (SO_3^{2-})	377.1		
	Residual Chlorine	330.1		
	pH (water)	150.1,9040,9045		
	Hydrazine	ASTM D1385		
Analyze within 24 hrs	Chromium (VI)	7196		
Analyze within 48 hrs.	Biological Oxygen Demand	405 1		
	Carbonaceous BOD (cBOD)	5210B		
	Color	110.2		
	MBAS/Surfactants	425 1		
	Nitrate by cadmium reduct.	353.2		
	Nitrite by cadmium reduct.	353.2		
	Orthophosphate by Spec	365.3		
	Nitrate by IC	300.0		
	Orthophosphate by IC	300.0		
	Nitrite by IC	300.0		
	Settleable Solids	SM2540F		
	Turbidity	180.1		
Filter 8-96 hrs after acidification	Potentially dissolved Metals	200.7		

Additional Tests:

Sample Number										
Date Sampled										
Time Sampled										

ATTACHMENT F

INTERNAL CHAIN-OF-CUSTODY FORM

Internal Chain-of-Custody *STL Denver*

STL Lot #	**Initial Receipt Date**	**Sample Custodian Initials**

Samples Stored in Sample Receiving Walk-In		
Sample #s	Location in Walk-In (or Satellite area for strict internal COC samples)	Matrix

Sample #s	Department	Test(s)	Matrix	Date/Time Out	Analyst Initials	Date/Time In	Analyst Initials

Satellite Area Sample Transfers								
Satellite Area		**Sample Numbers**	**Transfer Date**	**Storage Location**	**2nd Transfer Date**	**2nd Storage Location**	**Archive Date**	**Archived By**
Metals (aqueous, non-AFCEE)								
GC Volatiles	Water							
	Solid							
MS Volatiles	Water							
	Solid							

Transfers to Sample Archive					
Sample #s	Area Received From	Date Received	Archived By	Archive Storage Location	Disposal Date

Revised 01/17/01

Strict Internal Chain-of-Custody Required: ________

Controlled Document
Copy Number_______________

Implementation Date 01/17/01

STL-Denver

Interim SOP Change - This is a controlled document

SOP NUMBER: **DEN-QA-0003**

SOP TITLE: Sample Management and Chain-of-Custody
SOP SECTION(S) AFFECTED BY CHANGE: Attachment F
REASON FOR CHANGE(S): To accommodate Mixed Waste area
Change section *(Attachment F)* **from:** Old form **To:** New form including mixed waste area.
SUBMITTED BY/DATE: Jennifer Brown 01-05-01
APPROVED BY:
Jennifer Brown — **Technical Reviewer** — 01-09-01 **Date**
James S. Kelly, Health and Safety Coordinator — 01-09-01 **Date**
Larry Penfold, Quality Assurance Manager — 1-17-01 **Date**
Timothy M. O'Shields, Laboratory Director — 1/17/01 **Date**

Internal Chain-of-Custody *STL Denver*

STL Lot #	Initial Receipt Date	Sample Custodian Initials

Samples Stored in Sample Receiving Walk-In

Sample #s	Location in Walk-In (or Satellite area for strict internal COC samples)	Matrix

Sample #s	Department	Test(s)	Matrix	Date/Time Out	Analyst Initials	Date/Time In	Analyst Initials

Satellite Area Sample Transfers

Satellite Area		Sample Numbers	Transfer Date	Storage Location	2nd Transfer Date	2nd Storage Location	Archive Date	Archived By
Metals (aqueous, non-AFCEE)								
GC Volatiles	Water							
	Solid							
MS Volatiles	Water							
	Solid							
Mixed Waste	Water							
	Solid							

Transfers to Sample Archive

Sample #s	Area Received From	Date Received	Archived By	Archive Storage Location	Disposal Date

Strict Internal Chain-of-Custody Required: _______

ATTACHMENT G

INSTRUCTIONS IN THE USE OF THE INTERNAL CHAIN-OF-CUSTODY FORM

Instructions in the Use of the Internal Chain-of-Custody Form

The internal COC form (ICOC) has three sections. The top section records the Lot#, initial receipt date and custodian's initials. Also recorded in this section are the samples stored in the walk-in and their matrix. **When recording the matrix, use descriptors such as water, soil, or solid rather than the Quantims codes I or A.** The sample custodian is required to complete these sections during sample log-in.

The bottom portion of section 1 records the individual sample custody transactions for all samples stored in the walk-in. To complete this section, follow the instructions listed below:

1. **It is mandatory that each time a sample is removed from the walk-in, the analyst records it on the ICOC.**
2. When taking samples from the walk-in, complete each of the first six columns in the section.
3. When returning samples to the walk-in complete the date/time in and analyst initials fields.
4. **If any of the samples taken from the walk-in will not be returned, this also must be recorded on the ICOC. The reason for the non-return must be recorded in the date/time in field. Use the following codes to designate the reason:** ***C = sample consumed during analysis. B = sample broken during analysis. T/(second analyst's initials) = sample transferred to second analyst. Oth = other, described in detail on back of the ICOC.***
5. If a sample is transferred to a second analyst, it is the responsibility of the first analyst to record the transfer on the ICOC using the 'T' code described above. It is the responsibility of the second analyst to record the date and time returned to the walk-in on the ICOC.

Section 2 denotes sample transfers to the satellite areas.

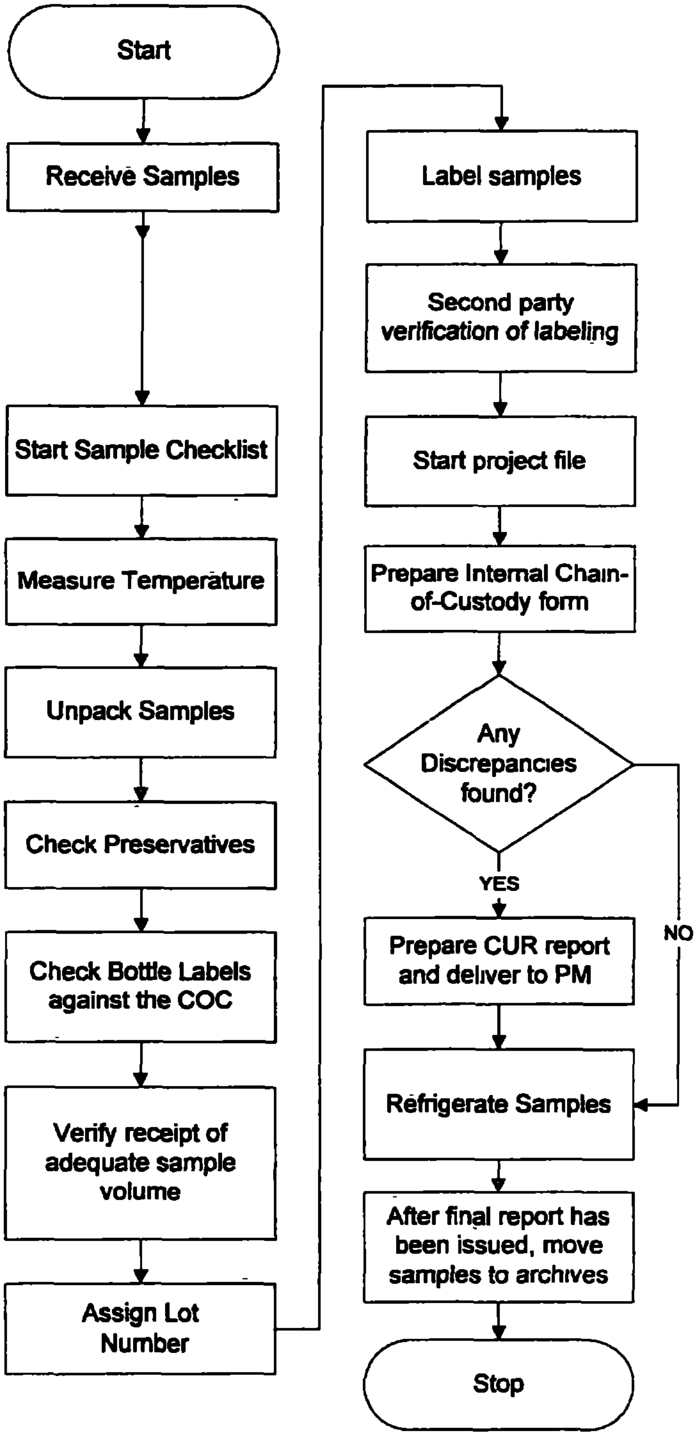
1. The sample custodian completes the first four columns for each applicable satellite area.
2. The specific storage location must be recorded (i.e., refrigerator ID rather than north refrigerator or shelf ID for metals samples).
3. The custodian then delivers a photocopy of the ICOC with the samples to each applicable group where it is retained until the samples are archived. **The original ICOC remains with sample receiving.**
4. When the samples are archived, the archive date and archive by fields are completed and the ICOC is then taken with the samples to the sample archive area. At this point the group copy is now an original record and must be retained and treated as such.
5. When strict internal chain-of-custody is required the area at the bottom of the form will be checked by sample receiving and each time a sample is handled in the satellite area it must be recorded using the area in Section 1.

Section 3 records sample transfers to the sample archive.

1. It is the responsibility of the area delivering the samples to the archive area to complete the first three columns, and it is the responsibility of the archive area personnel to complete the last three columns whenever samples are archived.
2. Archive area personnel are responsible for storing and archiving the completed ICOC forms in compliance with Quanterra guidelines.

ATTACHMENT H

FLOW CHART


Start
Receive Samples
Start Sample Checklist
Measure Temperature
Unpack Samples
Check Preservatives
Check Bottle Labels against the COC
Verify receipt of adequate sample volume
Assign Lot Number
Label samples
Second party verification of labeling
Start project file
Prepare Internal Chain-of-Custody form
Any Discrepancies found?
YES
NO
Prepare CUR report and deliver to PM
Refrigerate Samples
After final report has been issued, move samples to archives
Stop

STL - Denver
Condition Upon Receipt Anomaly Report (CUR)

Client: ____________________ Date/Time: ____________________

Lot No. ____________________ Initiated by: ____________________

Affected Samples COC# __________

Client ID	Lab ID	Analyses Requested

CONDITION/ANOMALY/VARIANCE (CHECK ALL THAT APPLY):

❑**COOLERS**
- ❑Not Received, No Chain of Custody (COC)
- ❑Not Received but COC(s) Available
- ❑Leaking
- ❑Other: ____________________

❑ **TEMPERATURE (greater than 6°C)**
- ❑Cooler Temp ____________________
- ❑Temperature Blank ____________________

❑**CONTAINERS**
- ❑Leaking
- ❑Broken
- ❑Extra
- ❑Without Labels
- ❑VOA Vials with Headspace __________ mm
- ❑Other: ____________________

❑**CUSTODY SEALS (COOLER(S)/CONTAINER(S)**
- ❑None
- ❑ Not Intact
- ❑Other: ____________________

❑**CHAIN OF CUSTODY (COCs)**
- ❑Not relinquished by Client; No date/time Relinq.
- ❑ Incomplete Information
- ❑Other: ____________________

❑ **CONTAINER LABELS**
- ❑Not the same ID/info as in COC
- ❑ Incomplete
- ❑ID COLLECTION ❑Time ❑Date ❑PRESERVATIVE
- ❑ Markings/Info smeared or illegible
- ❑Torn
- ❑Other: ____________________

❑ **SAMPLES**
- ❑Samples NOT RECEIVED but listed on COC ------ ❑will be noted on COC ❑ Client to send samples with new COC
- ❑Samples received but NOT LISTED on COC
- ❑Logged based on Label Information
- ❑Logged based on info from other samples on COC
- ❑ Logged according to Work Plan
- ❑Logged on HOLD UNTIL FURTHER NOTICE
- ❑ Other: ____________________
- ❑ Mislabeled as to tests, preservatives, etc.
- ❑Holding time expired
- ❑ Improper container used
- ❑Not preserved / Improper preservative used
- ❑ Improper pH __________ ❑ Lab to preserve sample
- ❑ Insufficient quantities for analysis

Comments: ____________________

Corrective Action:
- ❑ Client Informed: verbally on: ________ By: ________ : In writing on: ________ By: ________
- ❑ Sample(s) processed "as is". ____________________
- ❑ Sample(s) on hold until: __________ If released, notify: __________

Sample Control Supervisor Review: ____________________ Date. __________

Project Management Review: ____________________ Date: __________

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

3

3

HEALTH AND SAFETY PLAN

HEALTH AND SAFETY PLAN

Introduction

This Health and Safety Plan (Plan) applies to personnel who will potentially be exposed to ground water affected by creosote or coal tar constituents during the retrieval of ground water samples from active pumping wells, the GAC plant, monitor wells, and piezometers. This Plan has been designated to comply with, as a minimum, the requirements set forth in 29 CFR 1910.120, the OSHA standards governing hazardous waste operations. In no case may work be performed in a manner that conflicts with the intent of or the safety concerns expressed in this Plan.

Materials of Concern and Effects of Overexposure

The materials of concern which have been identified for this project are coal tar and creosote-related materials including naphthalene, other polynuclear aromatic hydrocarbons (PAH) and phenolic compounds.

Coal tar and creosote are typically irritating to the eyes, skin and respiratory tract. Acute skin contact may cause burning and itching while prolonged contact and poor hygiene practices may produce dermatitis. Prolonged skin contact with creosote must be avoided to prevent the possibility of skin absorption.

Naphthalene is a hemolytic agent which, upon overexposure to the vapor or ingestion of the solid, may produce a variety of symptoms associated with the breakdown of red blood cells. Naphthalene is also irritating to the eyes and repeated or prolonged contact has been associated with the production of cataracts.

Repeated exposure to certain PAH compounds has been associated with the production of cancer. Contact of PAH compounds with the skin may cause photosensitization of the skin producing skin burns after subsequent exposure to ultraviolet radiation.

Phenolics are generally strong irritants which can have a corrosive effect on the skin and can also rapidly penetrate the skin. Overexposure to phenols and phenolic compounds may cause convulsions as well as liver and kidney damage.

Hazard Assessment

<u>Initial</u>

Because of the relatively low vapor pressures associated with PAH compounds (generally less than 10^{-4} mm Hg at 20EC), they are not expected to present a vapor hazard. The most likely threat of exposure to these compounds will be via skin contact.

Action Limits

The American Conference of Governmental Industrial Hygienists (ACGIH) has established threshold limit values (TLV) for phenol and naphthalene at 5 and 10 ppm, respectively, as 8-hour time weighted averages (TWA). Based on these values, the action limits in Table 1 have been set. The lower limit of 5 ppm is based on the TLV for phenol while the upper limit of 50 ppm is based on a minimum protection factor of 10 for a half-mask, air purifying respirator.

TABLE 1

Action Limits for Air Contaminants

Limit	Persistent Concentration in the Breathing Zone	Procedure
Lower	5 ppm	Don respirators, step up monitoring
Upper	50 ppm	Stop work and back off from immediate work area until levels subside in the breathing zone

Response

When the PID yields persistent breathing-zone readings at or above the lower action limit, workers in the affected area will don respirators. Air sampling will continue on a more frequent basis. If readings are persistent at or above the upper limit, workers shall back off from the immediate work area until measured breathing-zone concentrations fall below the lower limit, at which time operations will resume and normal air monitoring will continue. If breathing zone levels do not fall below the upper limit, workers are to leave the work area and report the condition immediately to the City, the Engineer, or its representative. If necessary, engineering controls will be instituted to maintain vapor concentrations below the upper limit or arrangements will be made to upgrade to Level B protection.

Personal Protective Equipment

Personal protective equipment (PPE) will be donned, as necessary, based on the hazards encountered. Listed below is the PPE to be utilized during this project and the conditions requiring its use.

PPE

Coveralls - Polyethylene coated Tyvek if work involves contact with affected soil or groundwater

Boots - Chemical resistant type if work involves contact with affected soil or groundwater

Hard hat - When working in the vicinity of operating heavy machinery

Face shield - If splash hazard exists

Gloves - Nitrile for potential contact with affected soil or groundwater

Respirator - MSA Comfo II with GMC-H Cartridges if PID reading exceeds 5 ppm or if dust or odors become objectionable

Chemical safety goggles - If eye irritation occurs

Because of the carcinogenicity of certain PAH compounds, and because of the skin hazards associated with PAH and phenolic compounds, it is important that appropriate protective clothing be worn during work activities, which may involve the possibility of skin contact with affected soil or groundwater. As a minimum, the presence of visible creosote or coal tar-related material shall constitute evidence of affected soil or groundwater.

Health and Safety Training

Personnel covered by this Plan must have received appropriate health and safety training prior to their working on the site. Training will include:

Requirements for and use of respirators and PPE
Required personal hygiene practices
Requirements for employees to work in pairs
Proper material handling
Proper sampling procedures
Maintenance of safety equipment
Effective response to any emergency
Emergency procedures
Hazard zones
Decontamination methods
General safety precautions

A copy of the Standard Safety Procedures (Table 2) will be given to each worker covered by this Plan.

TABLE 2

Standard Safety Procedures

Employees are required to work in pairs
Wash face and hands prior to eating, smoking, or leaving the site
No smoking or eating is allowed in the work area during excavation or sampling activities
Wearing of contact lenses is not permitted in the work area
Contaminated material (e.g., Tyvek coveralls) must be properly disposed of before leaving the site
All work must be conducted in accordance with local, state and federal EPA and OSHA regulations, particularly 29

TABLE 2

Standard Safety Procedures

Employees are required to work in pairs
CFR 1910.120

Decontamination

Administrative procedures require hygienic practices consistent with work hazards. employees will be instructed in the training program on proper personal hygiene procedures.

Contaminated, reusable PPE, such as boots, hard hats, face shields and goggles, will be decontaminated prior to leaving the site. The decontamination procedure follows:

Rinse with water to remove gross contamination
Wash in Alconox or equivalent detergent solution
Rinse with clean water

Contaminated, disposable PPE, such as Tyvek coveralls and gloves will be placed in 55-gallon drums and stored while arrangements are made for disposal.

Respirators, if used, will be cleaned and disinfected after each day of use. The face-piece (with cartridge removed) will be washed in a hypochlorite (or equivalent) disinfecting solution, rinsed in warm water and air dried in a clean place.

Emergency Procedures

This Plan has been established to allow site operations to be conducted without adverse impacts on worker health and safety as well as public health and safety. In addition, supplementary emergency response procedures have bee developed to cover extraordinary conditions at the site.

General

All accidents and unusual events will be dealt with in a manner to minimize a continued health risk to site workers. In the event that an accident or other unusual event occurs, the following procedure will be followed:

> First air or other appropriate initial action will be administered by those closes to the accident/event. This assistance will be conducted so that those rendering assistance are no placed in a situation of unacceptable risk. In the event that a worker is caught in a trench collapse, call for emergency assistance immediately.
>
> All accidents/unusual events must be immediately reported to the Owner.
>
> All workers on site should conduct themselves in a mature, calm manner in the

event of an accident/unusual event, to avoid spreading the danger to themselves, surrounding workers and the community.

Responses to Specific Situations

Emergency procedures for specific situations are given in the following paragraphs.

Worker Injury

If an employee in an affected area is physically injured, Red Cross first-aid procedures will be followed. Depending on the severity of the injury, emergency medical response may be sought.

If the injury to the worker is chemical in nature (e.g., overexposure), the following first-aid procedures are to be instituted:

Eye Exposure - If affected solids or liquids get into the eyes, wash eyes immediately using large amounts of water and lifting the lower and upper lid occasionally. Obtain medical attention immediately.

Skin Exposure - If affected solids or liquids get on the skin, promptly wash the affected skin using soap or mild detergent and water. Obtain medical attention immediately when exposed to concentrated solids or liquids.

Inhalation - If a person inhales large amounts of a toxic vapor, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Obtain medical attention as soon as possible.

Swallowing - When affected solids or liquids have been swallowed, the Poison Control Center will be contacted and their recommended procedures followed.

Emergency Notification

In an extraordinary event that might be damaging to personnel or adjacent property, immediate notification of the proper emergency service will be required. The proper emergency service is determined by the nature of the emergency.

EMERGENCY NOTIFICATION

Fire Department....................911
Ambulance....................911
Police Department....................911
Methodist Hospital....................932-5000
Poison Control Center....................347-3141

OTHER CONTACTS

MPCA – Nile Fellows....................651-296-7299

EPA - Darryl Owens 312-886-7089
City of St. Louis Park - Scott Anderson 952-924-2557
- William Gregg 952-924-0117

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COMMUNITY RELATIONS PLAN

COMMUNITY RELATIONS PLAN

The Sampling Plan is to be completed in accordance with the Consent Decree-Remedial Action Plan for Reilly Tar & Chemical Corporation's St. Louis Park, Minnesota, N.P. L. Site. All community relations programs related to this work will be coordinated through the following agencies:

United States	Ms. Denise Gawlinski United States Environmental Protection Agency (312) 886-9859
State of Minnesota	Ms. Katherine Carlson Minnesota Pollution Control Agency (651) 297-1607
City of St. Louis Park	Ms. Lynn Schwartz City of St. Louis Park (952) 924-2521

Information necessary to conduct the Community Relations Plan will be provided by the City and Reilly Industries, Inc.